Dynamic Imaging with Phase and Absorption Contrast

by Les Butler
Department of Chemistry
Louisiana State University

- large user community from GeoX 2010 workshop:
  - fantastic research, great ideas, really nice people!!!
- personal research:
  - flame retardants in polymer blends
  - neutron tomography: co-champion of SNS VENUS project
  - LSU CAMD synchrotron tomography beamline: testbed?
  - IMA math workshop
- wish list for NSLS II imaging beamline(s)

GeoX 2010, New Orleans, Louisiana

~120 participants,
many are users of laboratory X-ray tomography
Advances in Computed Tomography for Geomaterials;
Alshibli, K. A.; Reed, A. H., Eds.; Wiley, 2010.

Highlights of GEOX10

http://www.cee.lsu.edu/geox2010/workshop/default.htm

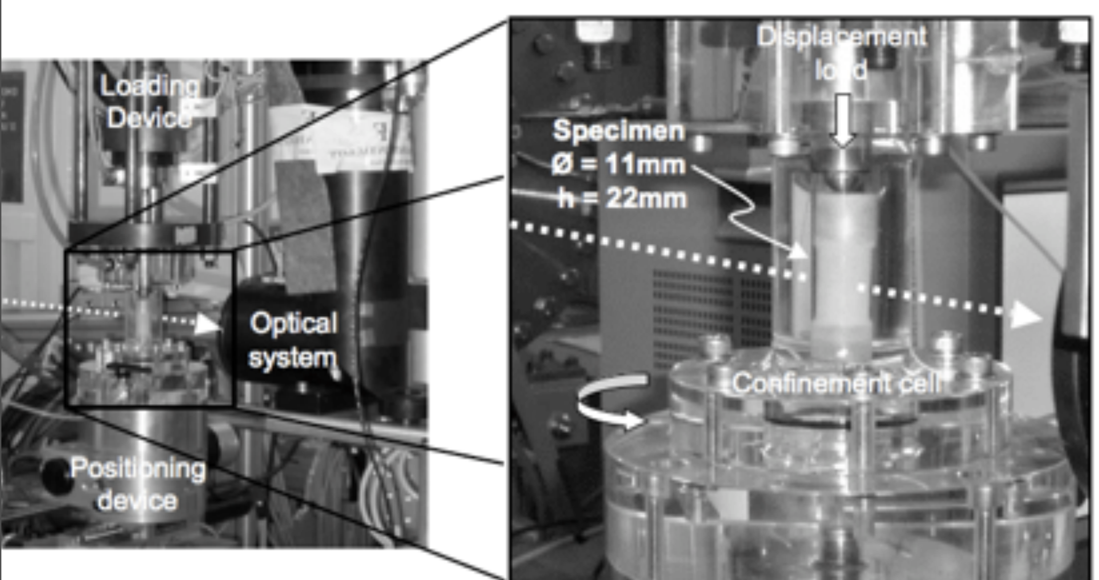
by Les Butler

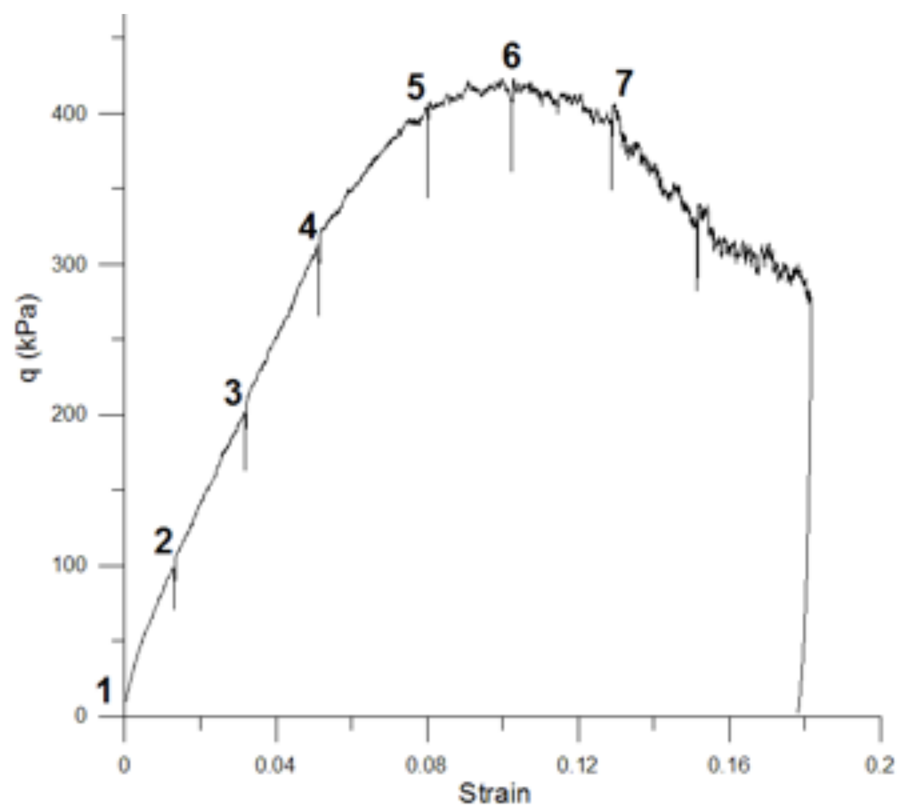
Topics

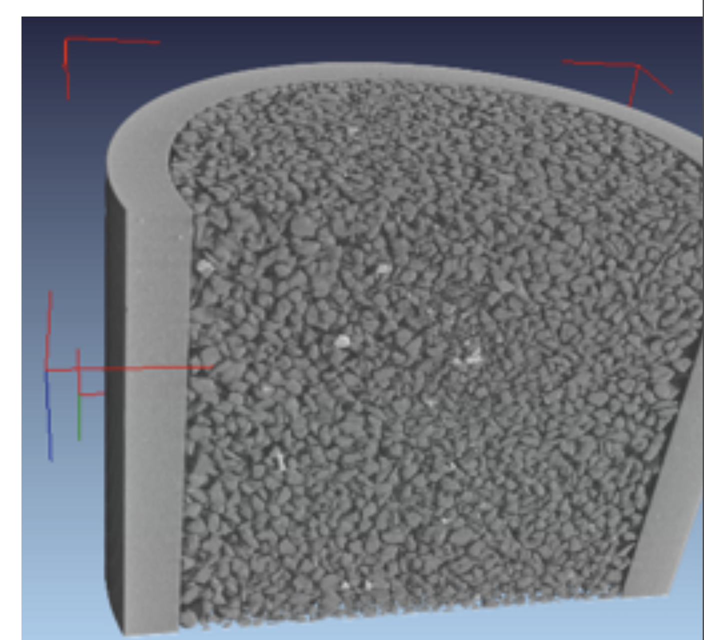
- sand grains: stress (shear banding)
- fluid flow particle tracking: over 100,000 particles (two methods)
- segmentation: 3 or more phases
- work flow
- image reconstruction: user GUI and beam hardening corrections
- digital image correlation: to align one 3D image to the next
- experimental chambers, sample holders
- crack imaging: current imaging results do not meet user's needs
- CO_2 sequestration; shale fracture; pressurized acid & rocks

p1 Gino Viggiani - sand grain mapping

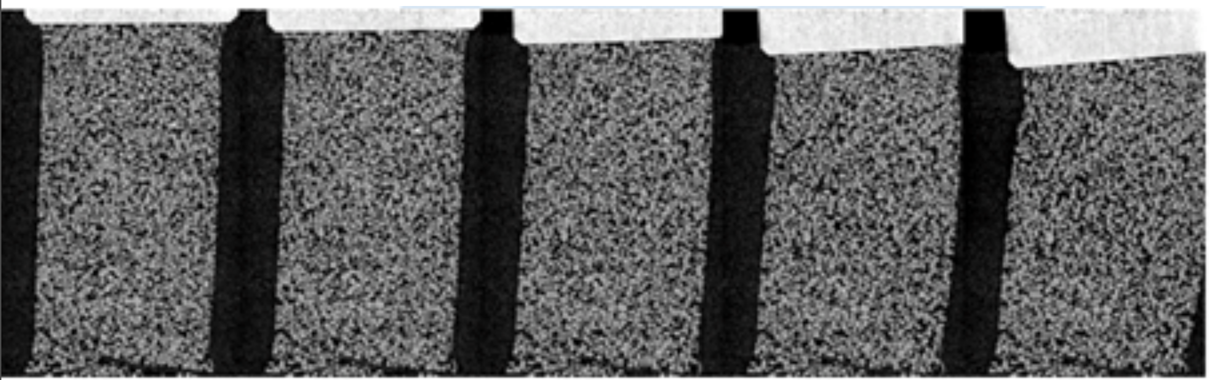
ABSTRACT. This paper presents a study of localized deformation processes in sand with grainscale resolution. Our approach combines state-of-the-art x-ray micro tomography imaging with 3D Volumetric Digital Image Correlation (3D V-DIC) techniques. While x-ray imaging and DIC have in the past been applied individually to study sand deformation, the combination of these two methods to study the kinematics of shear band formation at the scale of the grains is the first novel aspect of this work. Moreover, we have developed an original grain-scale V-DIC method that enables the characterization of the full kinematics (i.e. 3D displacements and rotations) of all the individual sand grains in a specimen. We present results obtained with both "continuum" and "discrete" DIC on Hostun sand, and a few preliminary results (continuum DIC only) recently obtained on ooid materials, which are characterized by spheroidal, layered grains.



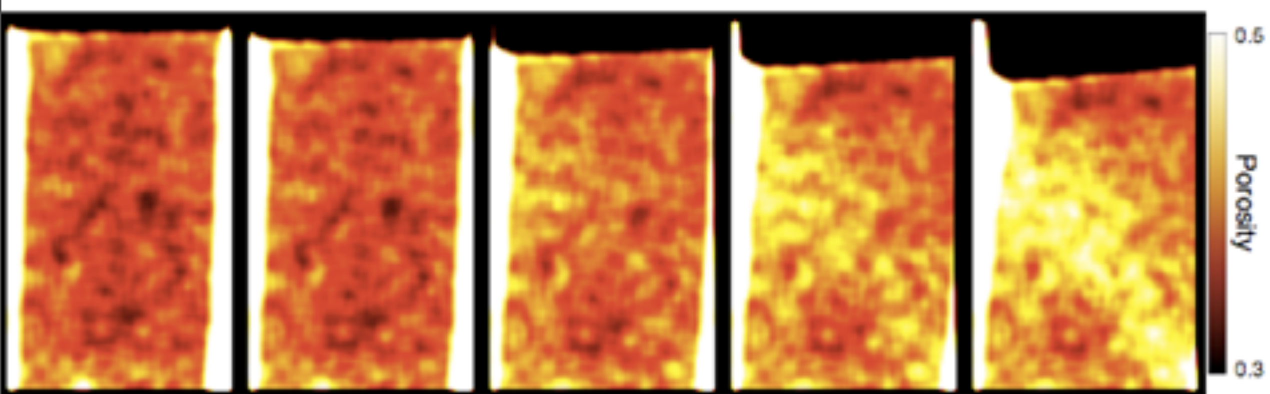




p1 Gino Viggiani - sand grain mapping



slices from tomography



porosity calculated from tomography showing “shear bands”

Grain tracking: If grain is about 20 voxels across (or volume ~5,000 voxels), their software can track the grain. Over 100,000 grains in sample were tracked across 8 pressure steps in the procedure called “3D discrete kinematics”. Following talk by Steve Hall (fresh results, not in book) described analysis of 100,000+ grains in terms of grain-to-grain contacts (coordination number). He is searching for best way to present the huge amount of data.

My question: How to visualize correlated and anti-correlated grain rotation?

p59 E. M. Charalampidou - digital image correlation, the odd Fig 2

ABSTRACT. In this work we employ x-ray tomography, 3D digital image analysis and 3Dvolumetric digital image correlation techniques to characterize localized deformation phenomena in sandstone. The specimens considered have been deformed in triaxial compression under a range of confining pressures (20-190MPa). Shear or compaction bands were observed, at low and higher confinement respectively. X-ray tomography images have been acquired before and after loading (unconfined) at different spatial resolutions (30 and 90 µm voxel size). The combination of both x-ray tomography and 3D DIC provides insights into the geometry and mechanisms of the localized features.

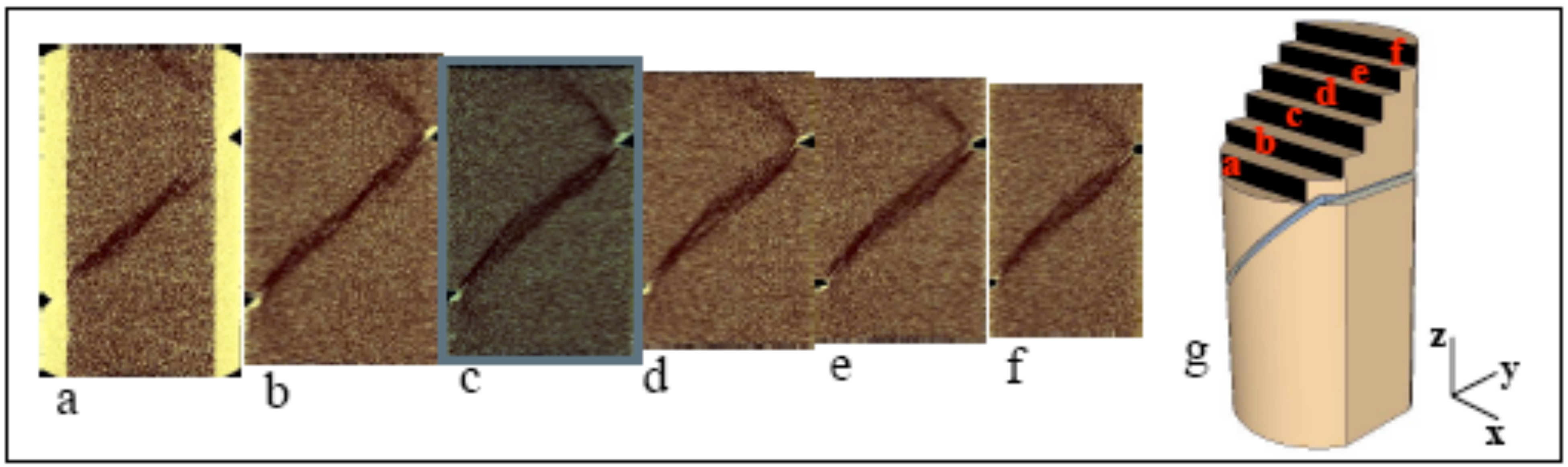
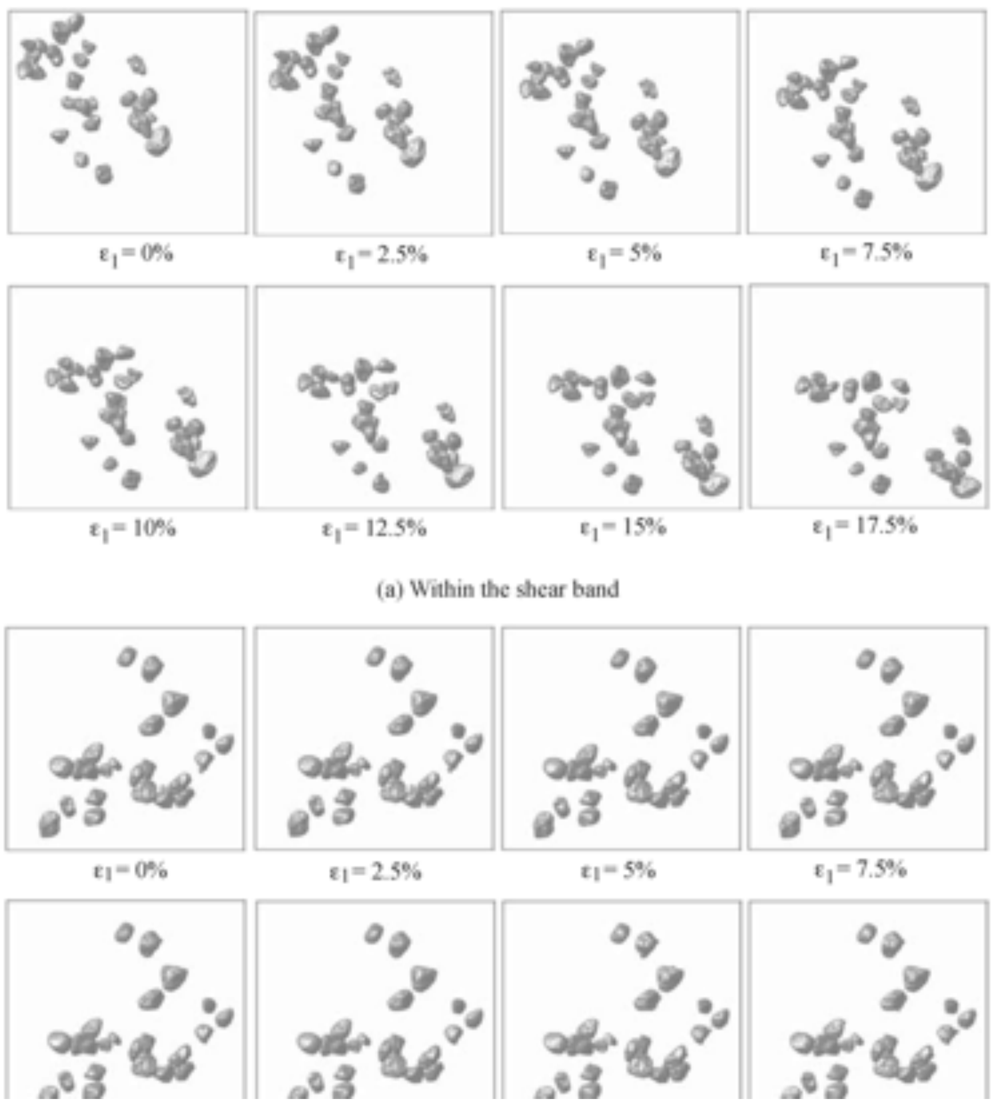


Figure 2. *Images from a to f represent sequence of views (x-z plane) through the image (y axis) showing the 3D structure of the shear band in VEC4 Vosges specimen (g)*

Shear band in this sandstone is remarkably planar. Also, plane connects two notches on side of sample chamber. Question: How do the two stress points communicate with each other to yield a single planar shear band?

ABSTRACT. Granular particles experience sliding and rolling as they are sheared. X-ray Synchrotron Microtomographic (SMT) was used to acquire 3D scans of a triaxial specimen of sand at eight axial strain levels. The specimen measures 9.5 mm in diameter and 20 mm in height. Several particles within and outside the shear band were identified and tracked as shearing progressed. The analysis reveals that sliding of particles within the shear band is much more significant than particles outside the shear band. Particles within the shear band continue to rotate throughout the experiment while particles outside the shear band exhibit insignificant rotation.



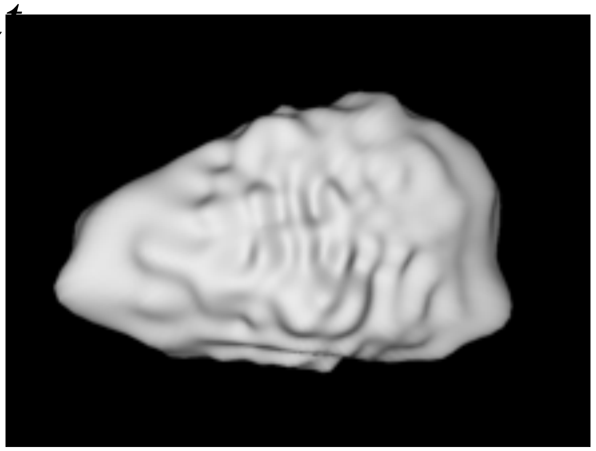
Student manually traced some dozens of grains in progression of 3D views and made short movie of grain translation and rotation. It's almost enough data to see vortices and correlated grain rotation, but needs much more work.

p93 Garboczi - spherical harmonics

ABSTRACT. Quantifying the shape of particles in three dimensions (3D) is important in particle technology. In concrete, the 3D shape of particles like sand, gravel and cement is of great interest for applications including suspension rheology, mechanical properties and realistic microstructure models. When particles are classified as star-shaped, a weaker condition than convexity, a combination of x-ray computed tomography (CT) and spherical harmonic series analysis can quantitatively describe their 3D shape. Since this analysis results in an analytic function for the particle's surface, one can perform almost any kind of volume or surface integral and so compute many geometrical properties. This paper reviews how 3D particle shape can be measured and analyzed and gives examples for the classes of particles found in construction. Some data will also be given on how particle shape can influence particle size measurement.

$$r(\theta,\phi) = \sum_{n=0}^{\infty} \sum_{m=-n}^{n} a_{nm} Y_{nm}(\theta,\phi)$$

<= spherical harmonics =>



Requires "star-shaped" structure (no voids or folds). Powder Technology **166**, 123 (2006) Apply to 3D after binarization, watershed. He is quantifying on the order of 100,000 particles with spherical harmonics. We are meeting April 7 and he will show me how to use his software.

p108 Kutay - matlab works well

ABSTRACT. In this paper, image processing and analysis methods are presented to extract the individual aggregate properties from the x-ray CT images of asphalt specimens. First, a technique was presented (and validated) to segment the clustered aggregates due to elevated pixel intensities near the aggregate-to-aggregate contacts in the image. Once the aggregates were segmented, aggregate-to-aggregate contact points, 3D orientation and segregation of aggregates were analyzed for asphalt specimens compacted at different levels. KEYWORDS: x-ray CT, aggregate, asphalt mixture, image processing

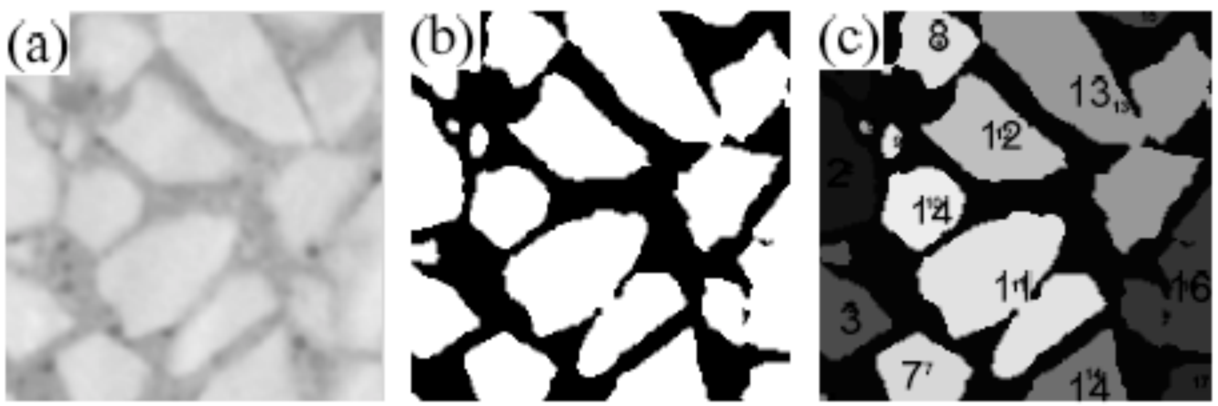


Figure 1. *Illustration of traditional binary thresholding and labeling; (a) original grayscale image, (b) thresholded binary image, and (c) labeled image*

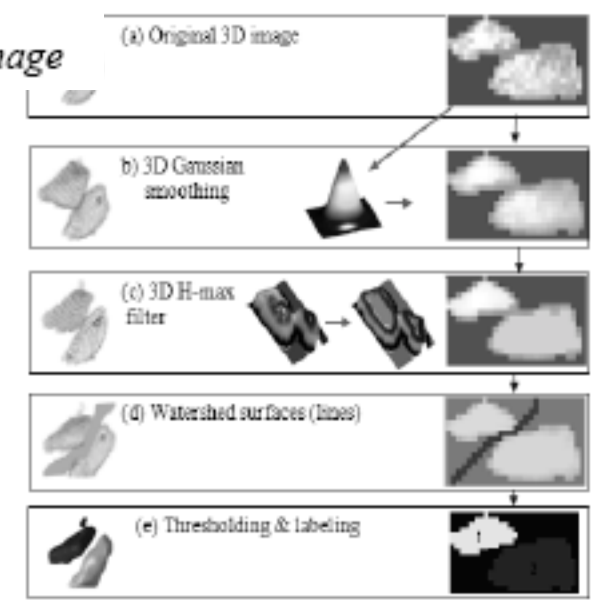


Figure 2. *Image processing steps for separation of aggregates*

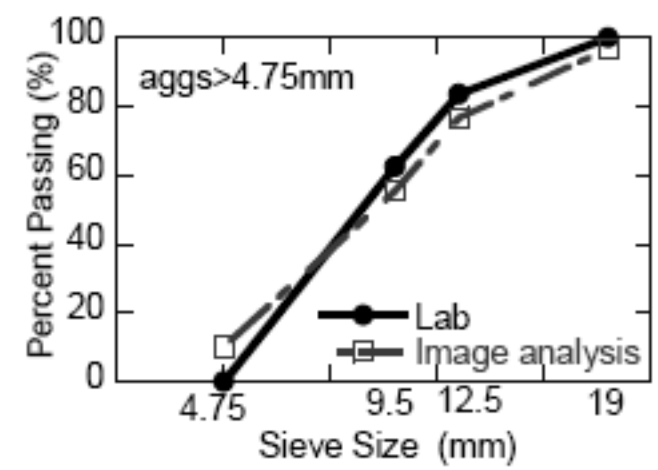


Figure 3. *Original and image-based back-calculated gradations*

p254 Bhattad - segmentation

ABSTRACT. X-Ray Computed Tomography (XCT) is an important tool to study porous-media microstructure and fluids present within the void space. In the presence of multiple fluid phases (e.g. air-water in soil science or oil-water-gas in petroleum engineering), the contrast between the fluid phases becomes important for accurate image segmentation. In some cases (e.g. a white light source or low flux), it is not possible to illuminate one of the fluid phases. The result is then a single image containing multiple phases that may contain overlapping peaks of the fluid phases due to little difference in the absorption coefficients. Building upon work done in medical-image-processing research, we have adopted a nonlinear anisotropic diffusion technique to remove noise from the XCT image that also leads to improved peak separation in the image histogram. The noise-free image is the then segmented using indicator kriging and the results are compared with segmentation results obtained using absorption-edge imaging.

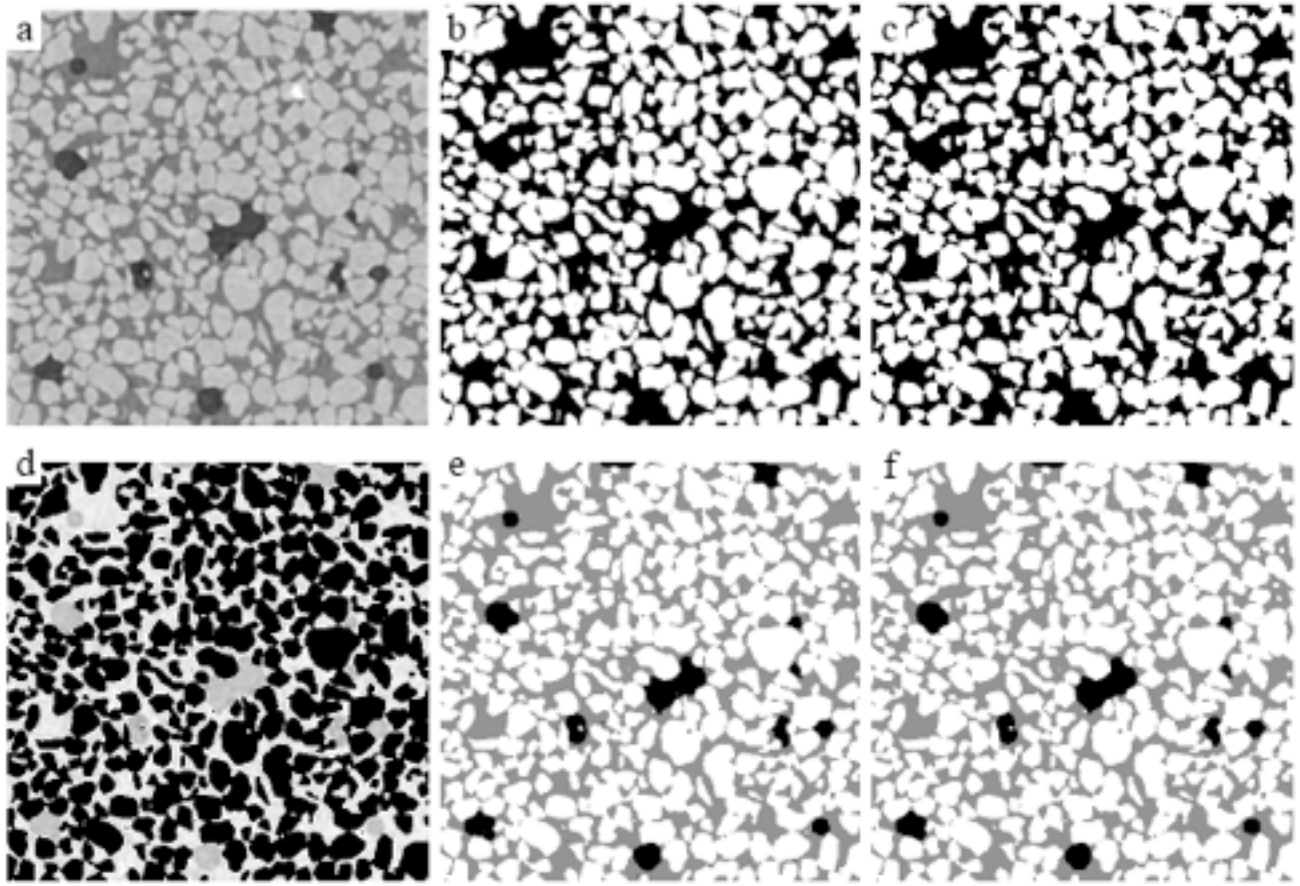
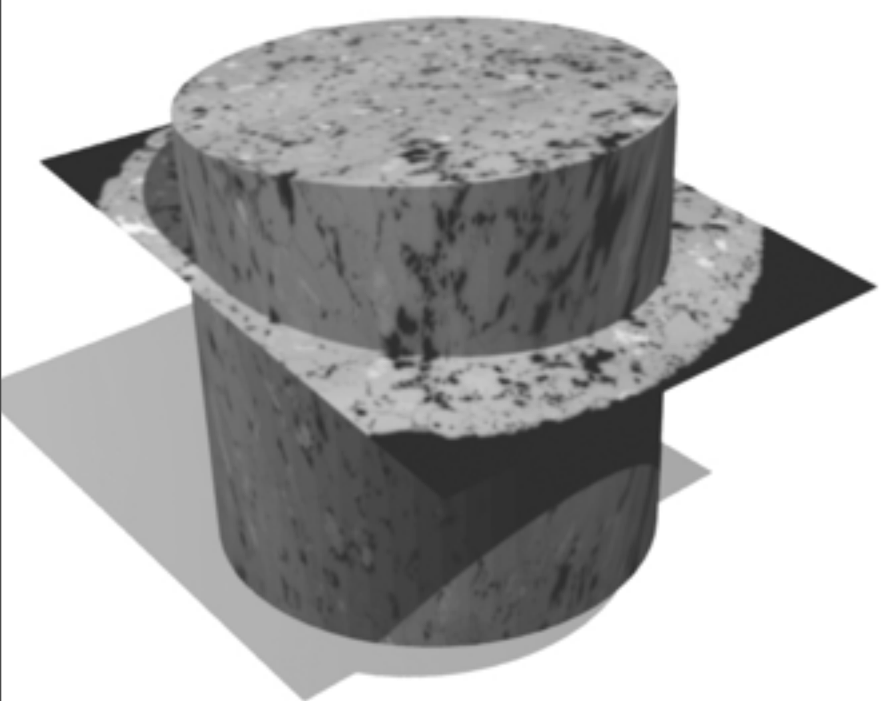


Figure 4. Section of the image showing the Indicator Kriging segmentation for 3 phase image. **(a)** Noise free below-edge image, **(b)** Partially thresholded image: White-solid, Black-Rest of the image containing Oil and Water, Gray-Unassigned voxels, **(c)** Segmented image showing solid (White) and rest of the image (Black), **(d)** Solid subtracted from the clean grayscale image, **(e)** Fully segmented three phase image using below edge image only white- Solid (57.508%), Gray-Water (35.436%) and Black-Oil phase (7.056%) and **(f)**Fully segmented three phase image using both above and below edge images, white-Solid (57.508%), Gray-Water (35.395%) and Black-Oil phase (7.097%).

p238 Senden - the ANU group



Procedure:

1) perform tomography
2) cut and polish the sample. Cut must be flat.
3) image with SEM. Work through the SEM image distortion problems.
4) register SEM image into the 3D volume
5) use SEM QEM-SCAN software to identify minerals in the SEM image
6) transfer that mineral segmentation information into the 3D volume

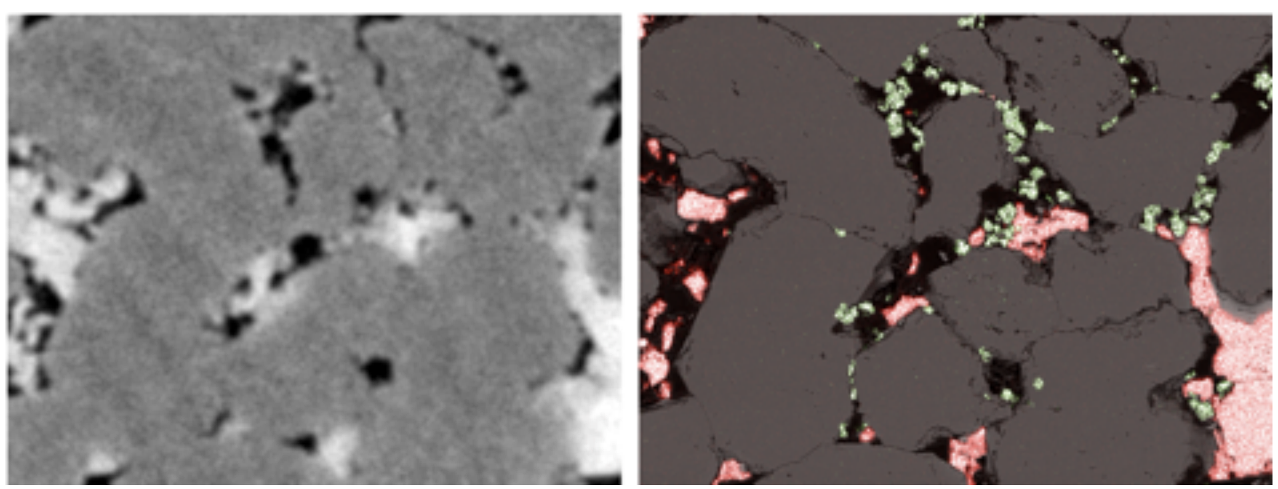


Figure 13. *A 300 µm wide tomographic slice from the siliclastic above registered with a mineralogical map from SEM for dolomite (green) and anhydrite (red). Note that the dolomitic phase on the left is often poorly distinguished from the quartz phase owning to small size. Without registration the difference between surface roughness and a distinctly different phase can be difficult to resolve.*

p336 Sugawara - tracers in fluid flow

ABSTRACT. X-ray Computed Tomography is successfully applied to the visualization of water flow in rock, and to the quantitative determination of the intrinsic permeability under various effective confining pressures. In a specific pressure vessel, one-dimensional water permeation test is performed. The transport diffusion phenomena of tracer is visualized and analyzed so as to evaluate the mean pore velocity of water in rock under the saturated condition, by utilizing the subtraction of CT images along with the data stacking technique. The intrinsic permeability is determined, based upon the Darcy's law. Case example clarify that the traceraided x-ray CT is a promising tool available for the evaluating the intrinsic permeability influenced by the effective confining pressure.

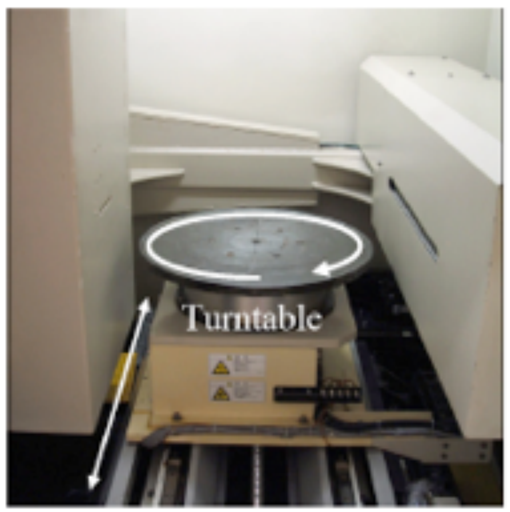


Figure 1. *X-ray CT scanner*

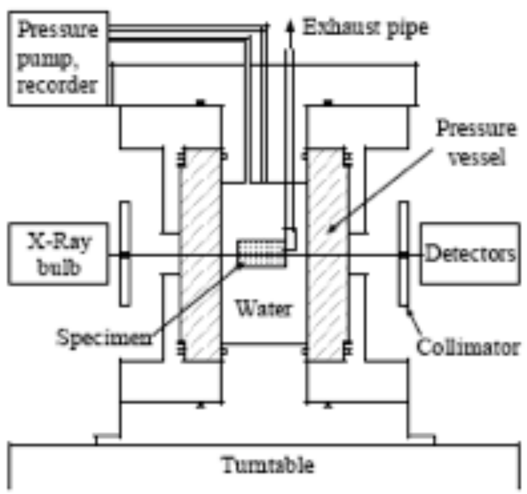


Figure 2. *Pressurization system*

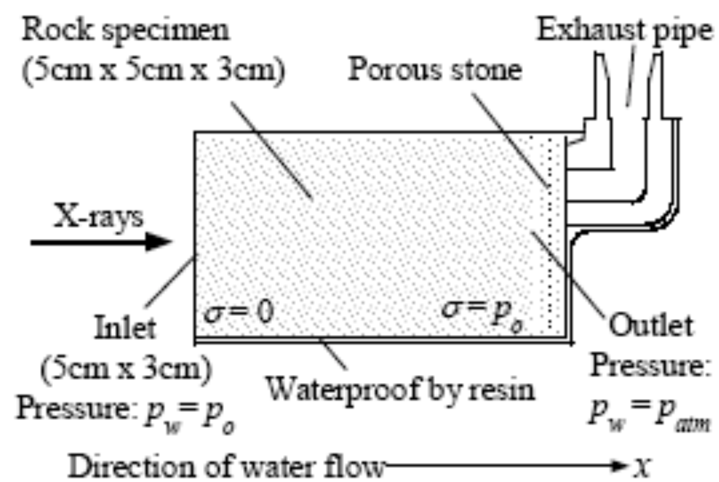
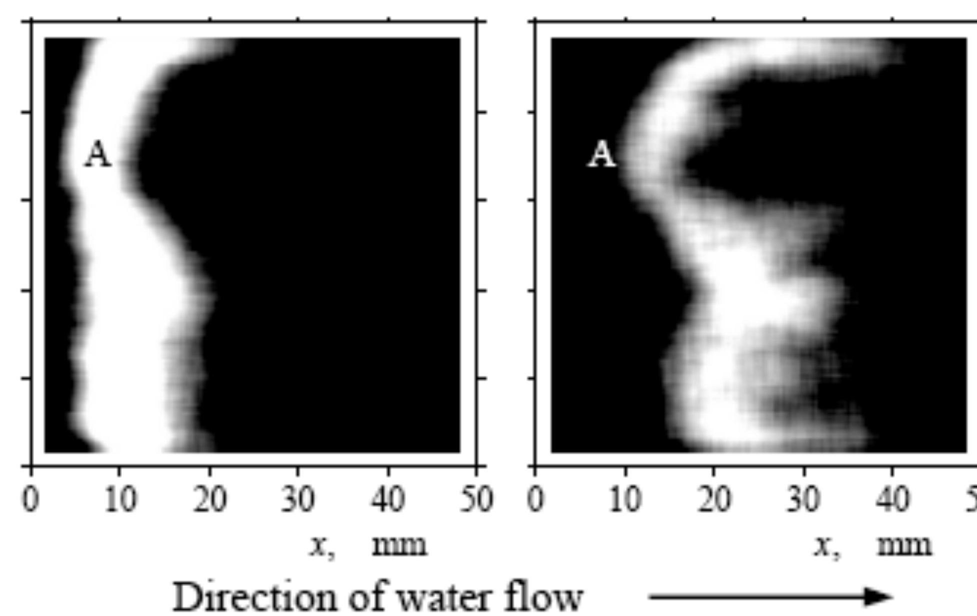


Figure 4. *Side view of specimen*



p230 Karpyn - two phase flow

ABSTRACT. Capillarity, gravity and viscous forces control the migration of fluids in geologic formations. However, experimental work addressing the impact of injection flow rate in fractured core samples is limited. Understanding how injection flow rate affects fracturematrix transfer mechanisms and invasion front evolution in fractured geomaterials are of crucial importance to modeling and prediction of multiphase ground flow. In this study, we monitor and analyze transfer mechanisms in a rock sample with a single tensile horizontal fracture using medical X-ray computed tomography. The impact of different injection rates on the resulting fluid recovery and saturation maps is evaluated through visual and quantitative analyses. Results from this investigation provide a comprehensive set of data for the validation of numerical models and strengthen fundamental understanding of multiphase flow in fractured rocks.

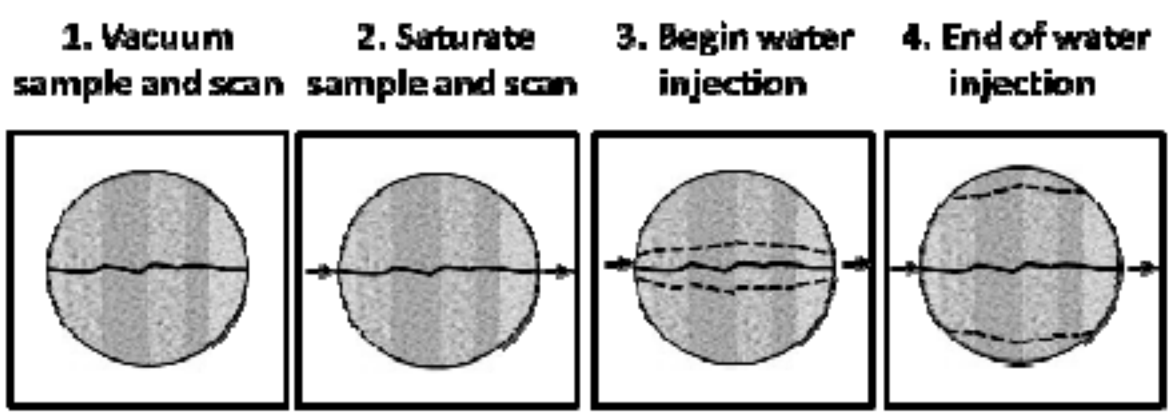


Figure 3. *Experimental procedure and CT scanning sequence*

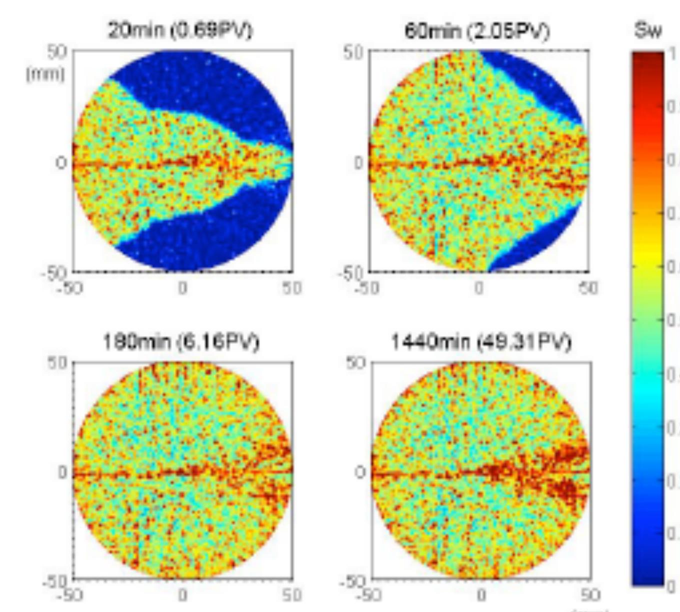


Figure 5. *Sequence of water saturation maps obtained from CT scanning at 40 mL/hr water injection rate*

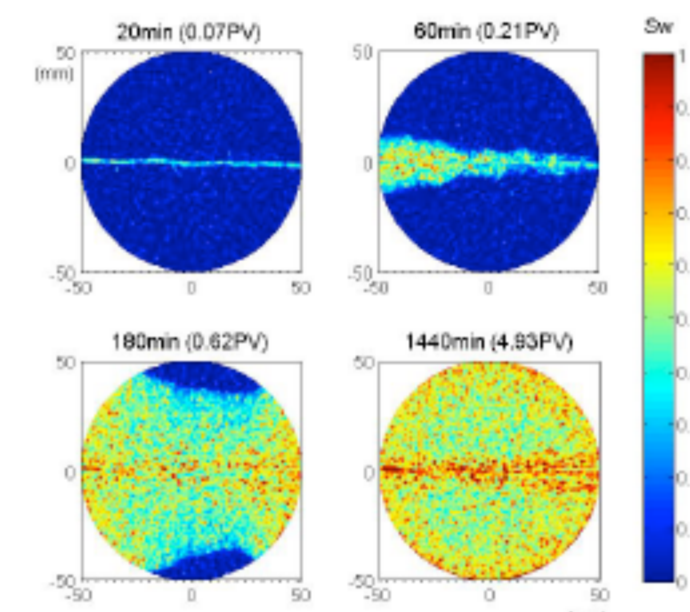
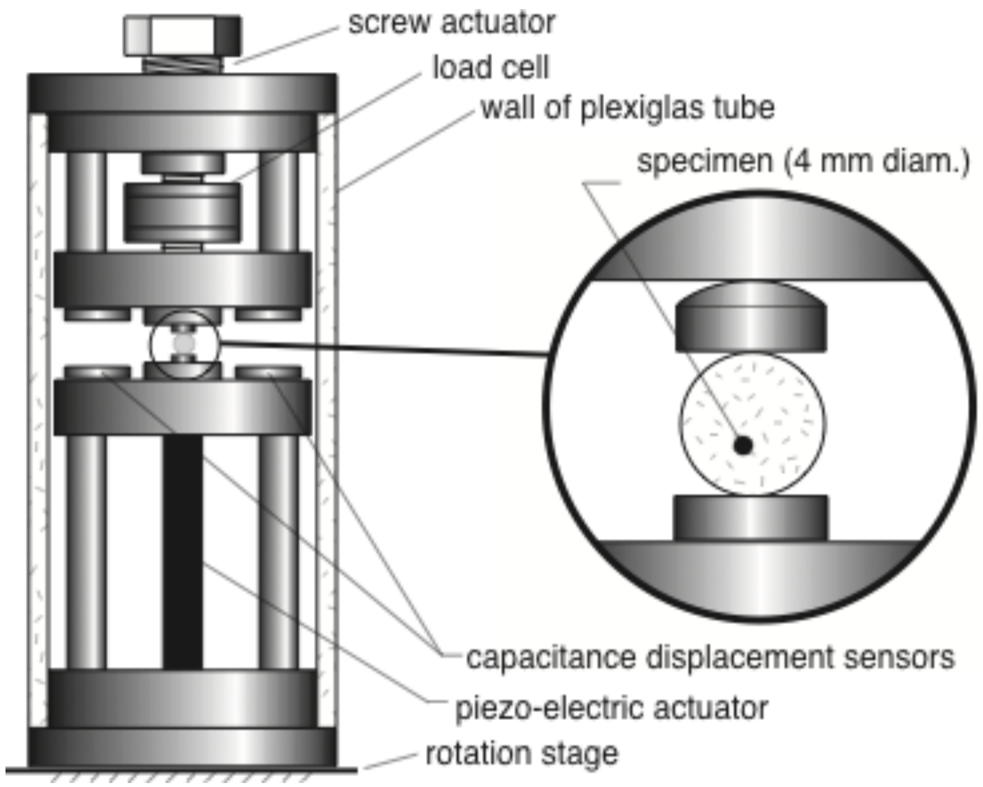


Figure 6. *Sequence of water saturation maps obtained from CT scanning at 4 mL/hr water injection rate*

p117 Landis- imaging and discrete element modeling

ABSTRACT. This paper describes a collaboration where synchrotron-based x-ray CT images of micromechanical experiments on cement-based composites are used to develop discrete element computational models of the material. Preliminary results are presented showing how 3D images are used to create computational models, as well as the resulting simulations from those models. The results show a good qualitative agreement between simulations and experiments, opening a door that will allow us to establish previously difficult-to-characterize properties such as cement-aggregate interfaces



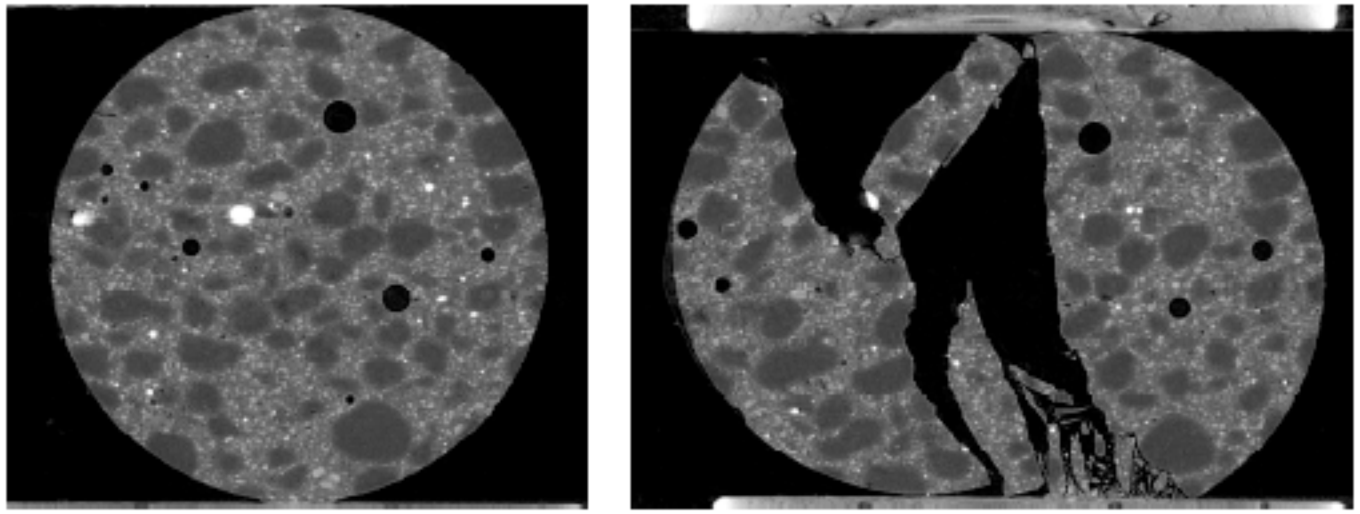
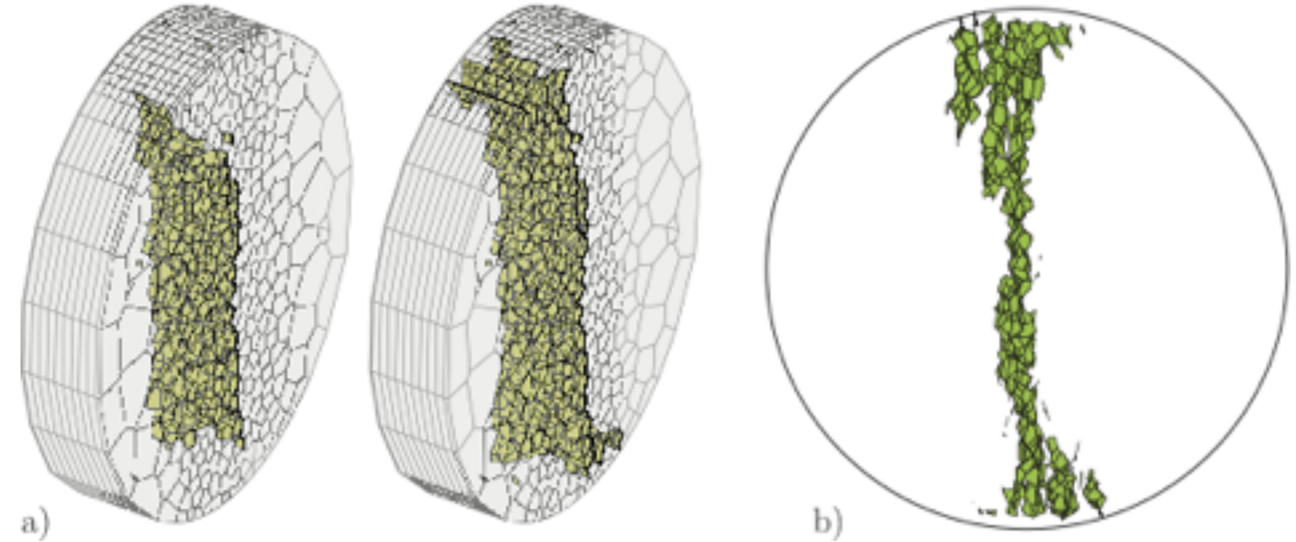
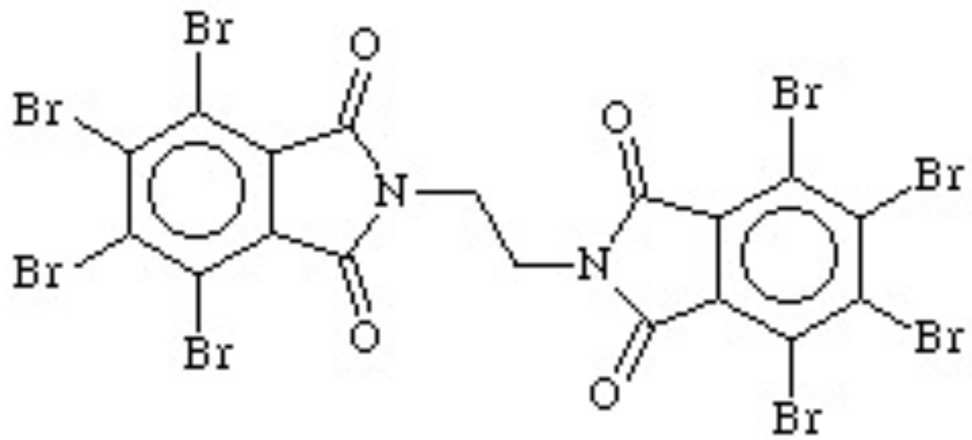
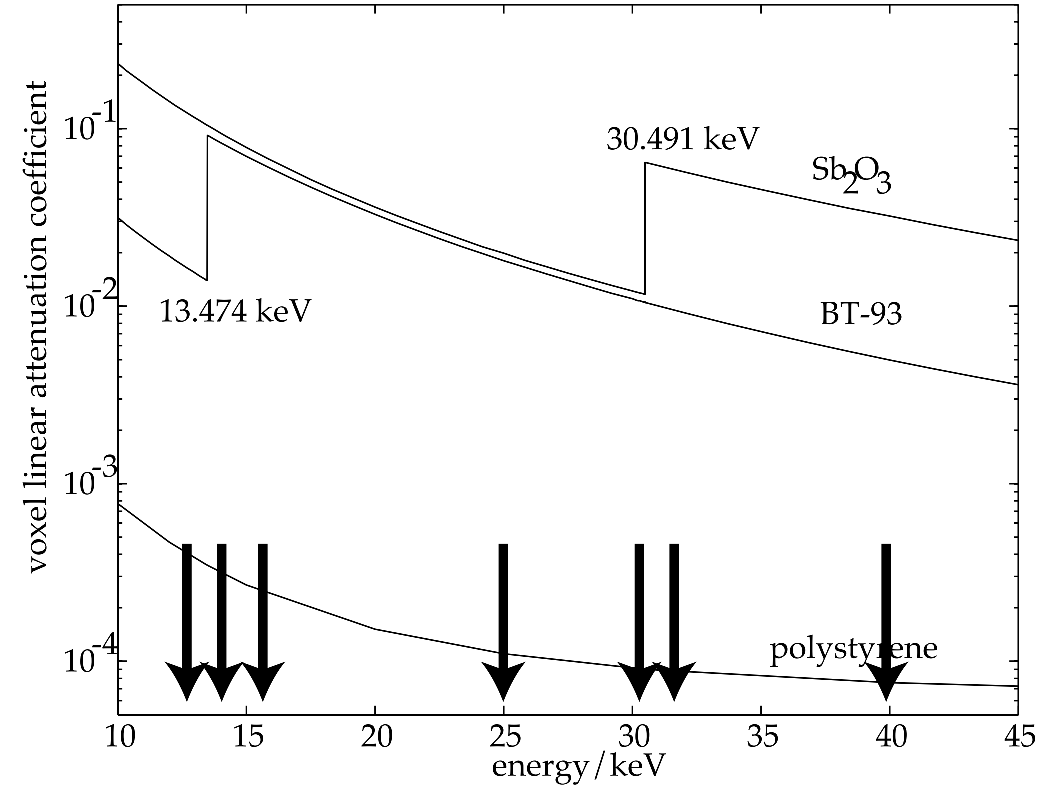


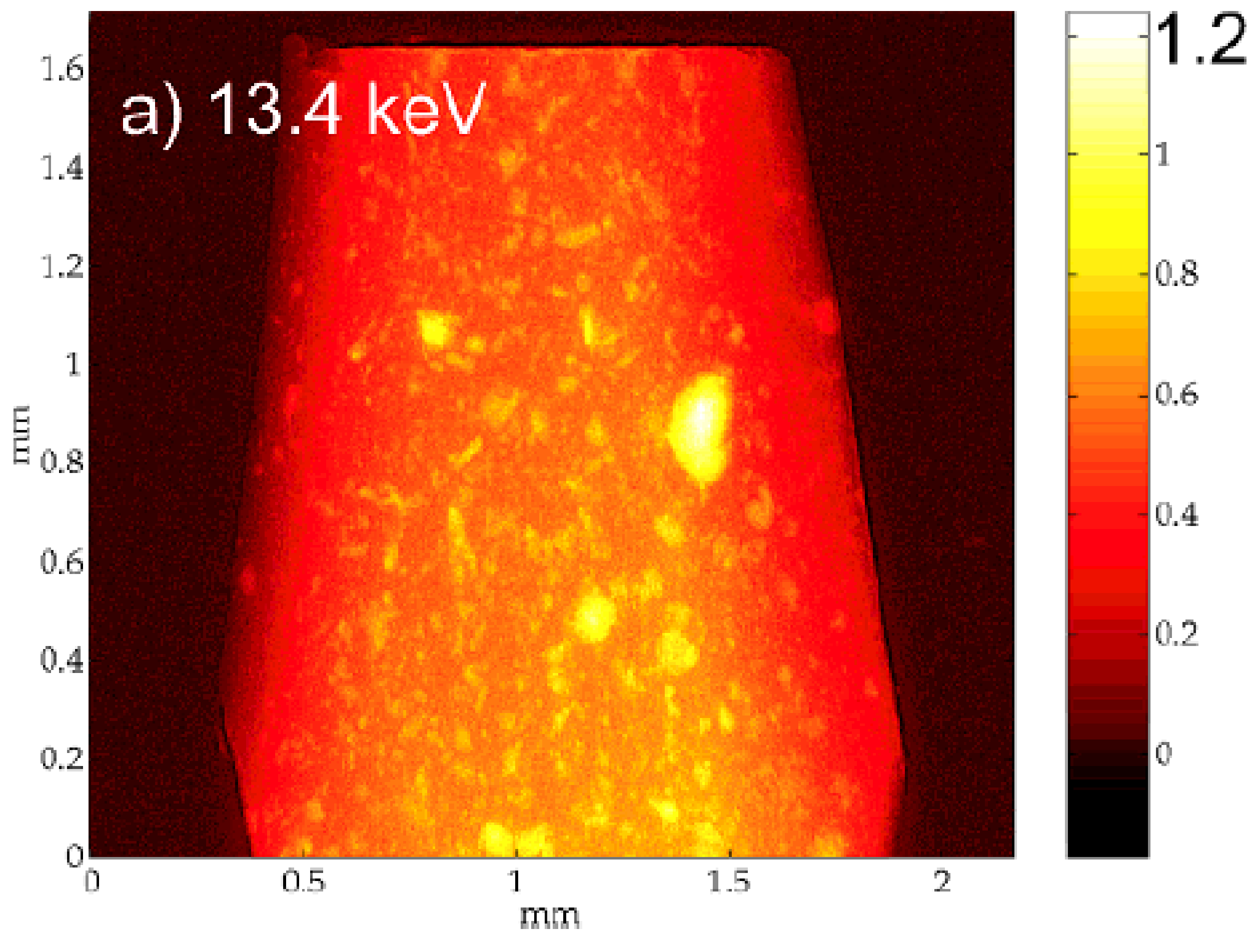
Figure 2. *Vertical slice images illustrating split cylinder fracture. Cylinder is 4 mm diameter. Note that due to non-uniform deformation, not all features in the damaged specimen image (right) appear in the same plane as the undamaged image (left)*



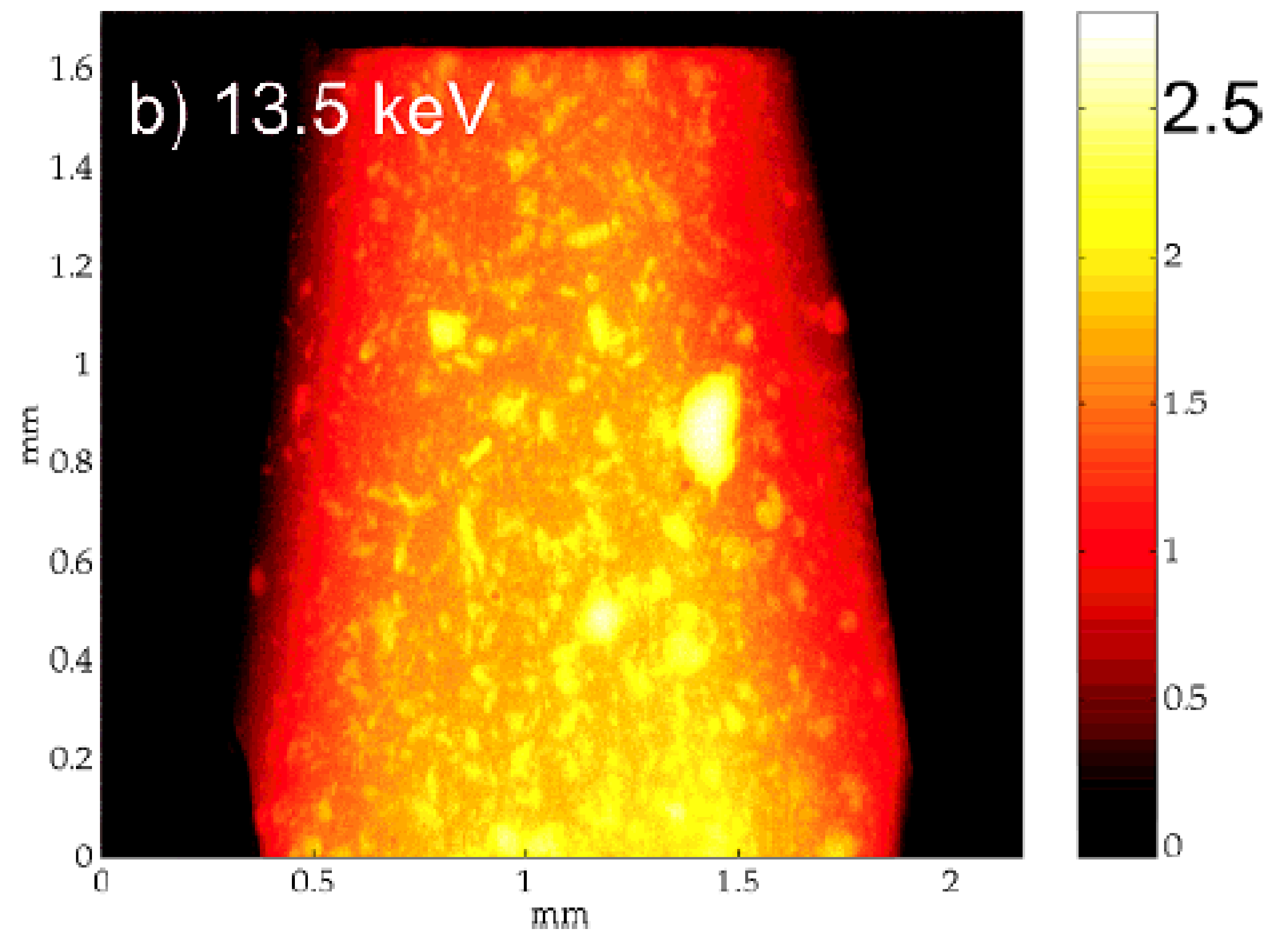


$$\mathrm{Br} + 13.474\ \mathrm{keV} \rightarrow \mathrm{Br}^{+} + e^{-}$$

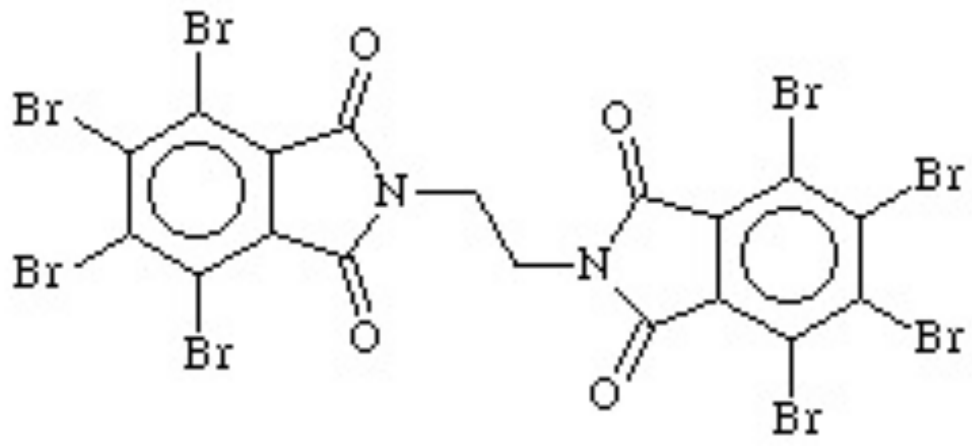




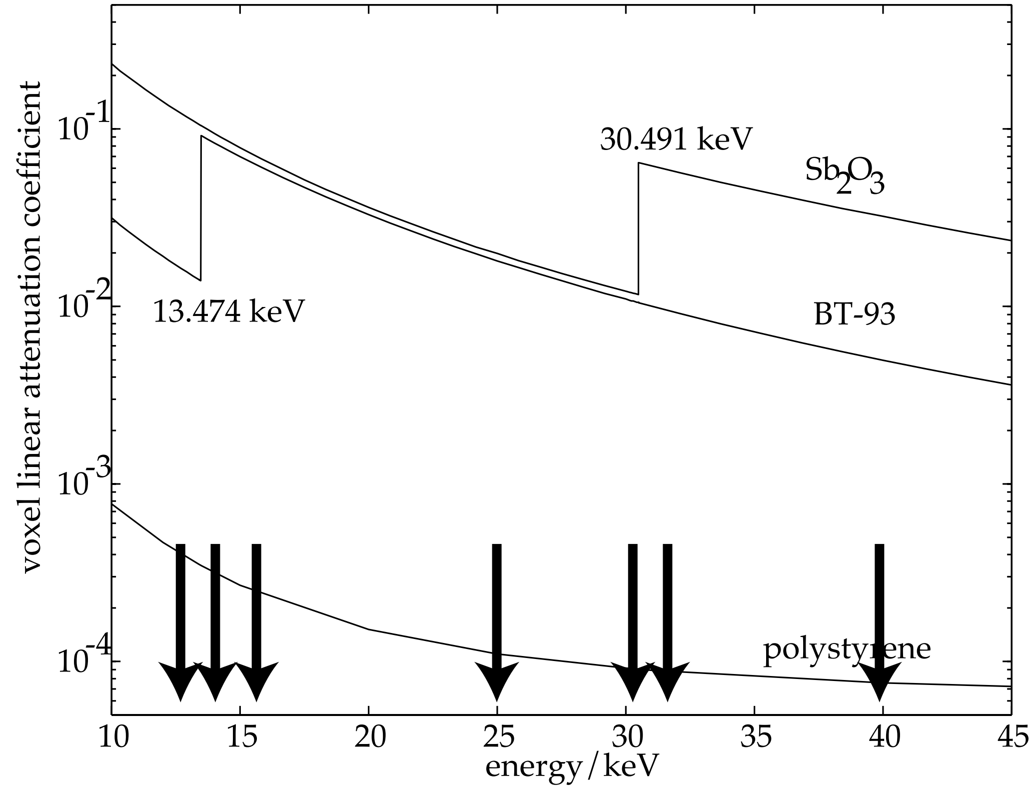
below K-edge

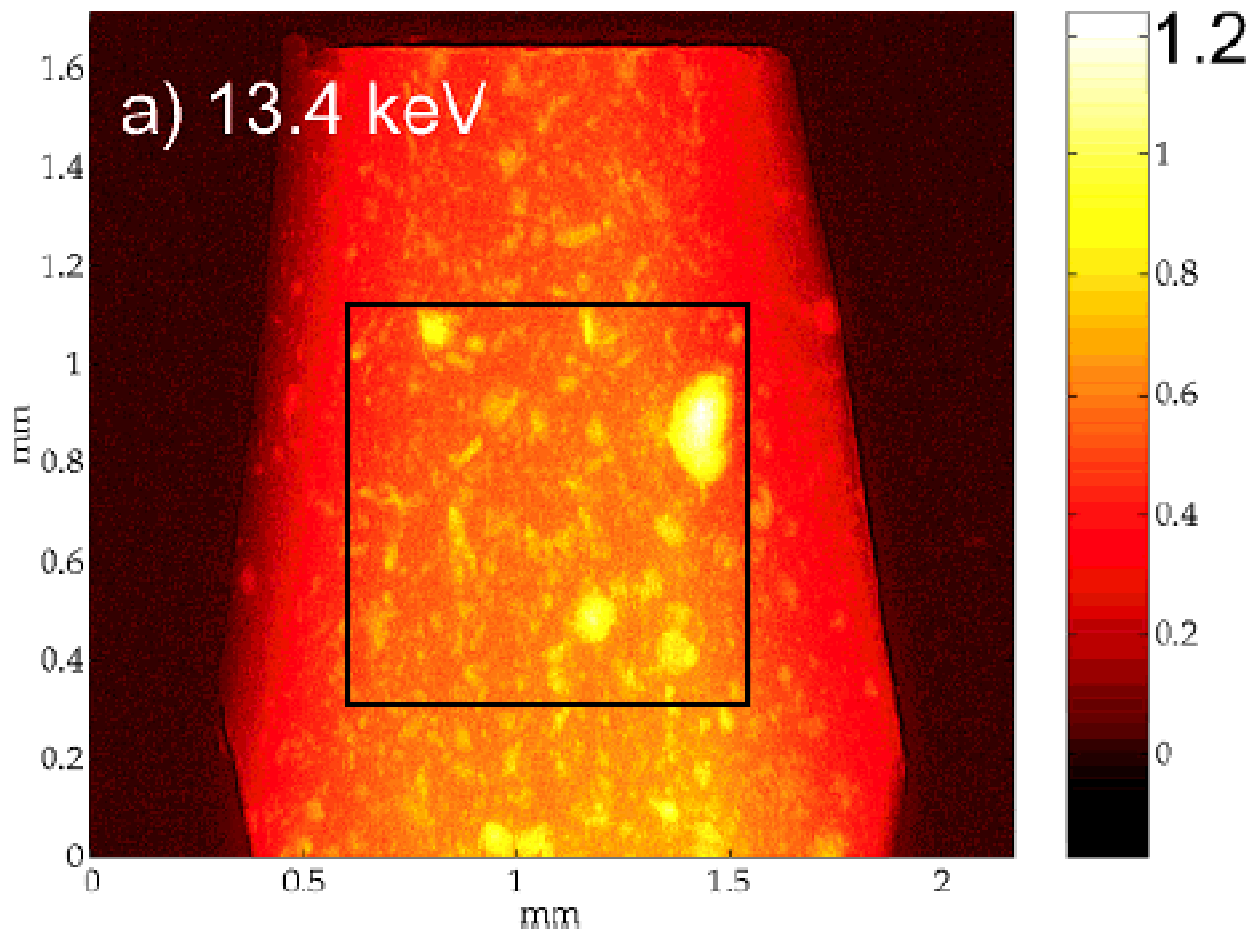


above K-edge

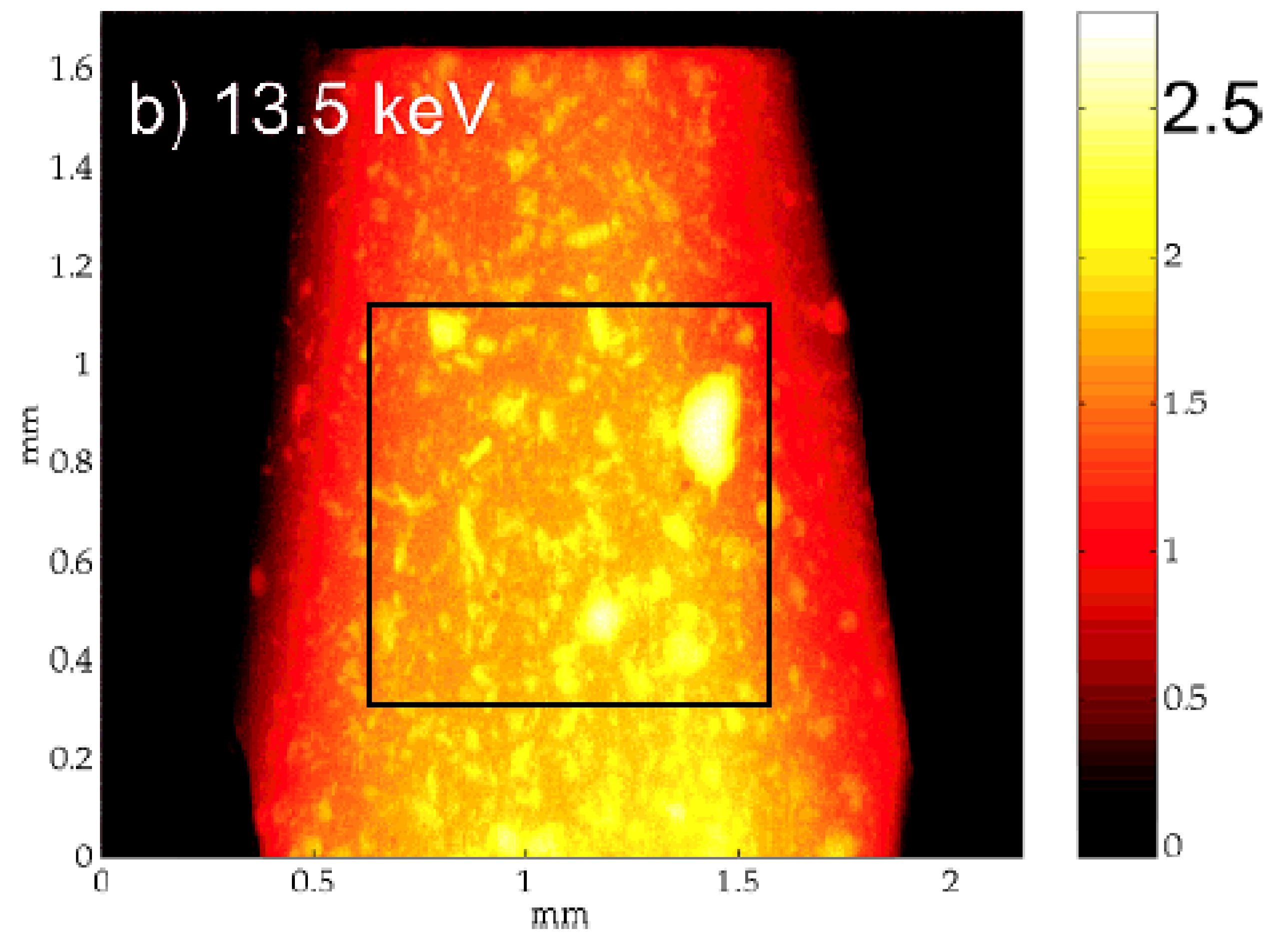


$$Br + 13.474\ keV \rightarrow Br^{+} + e^{-}$$



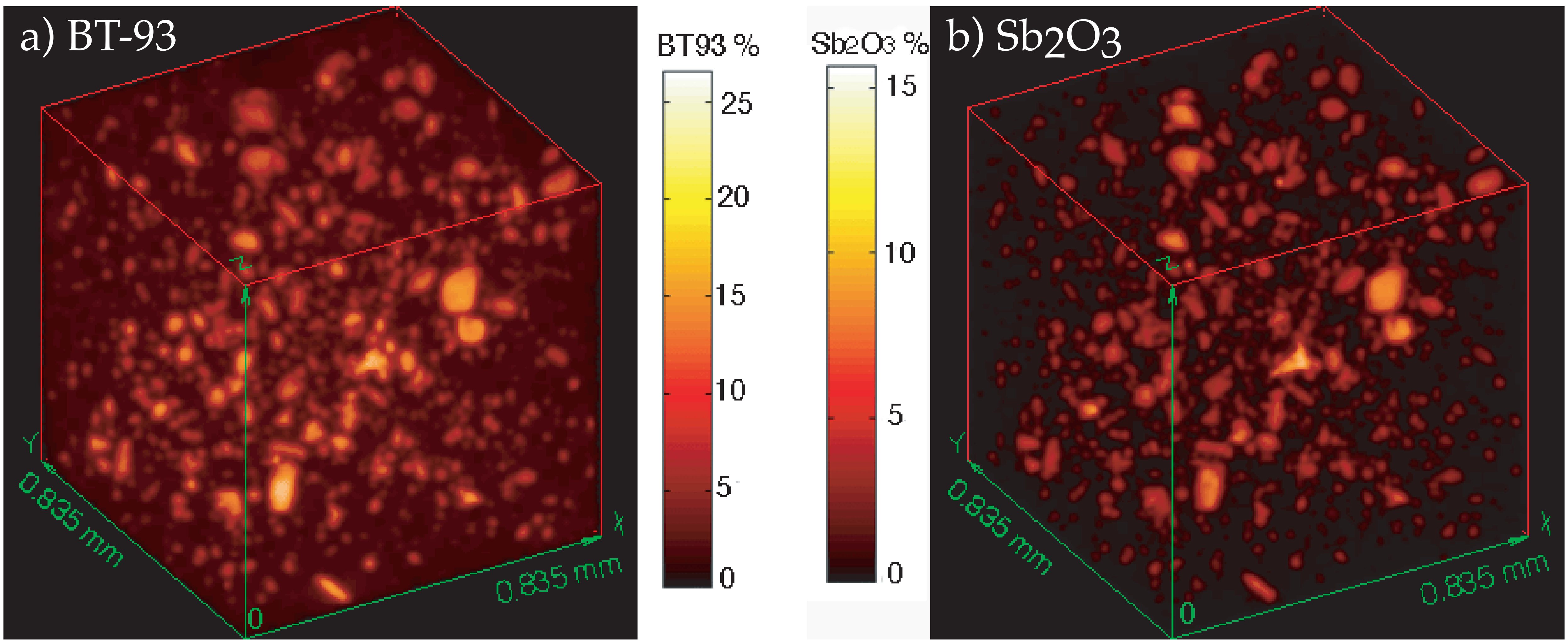


below K-edge



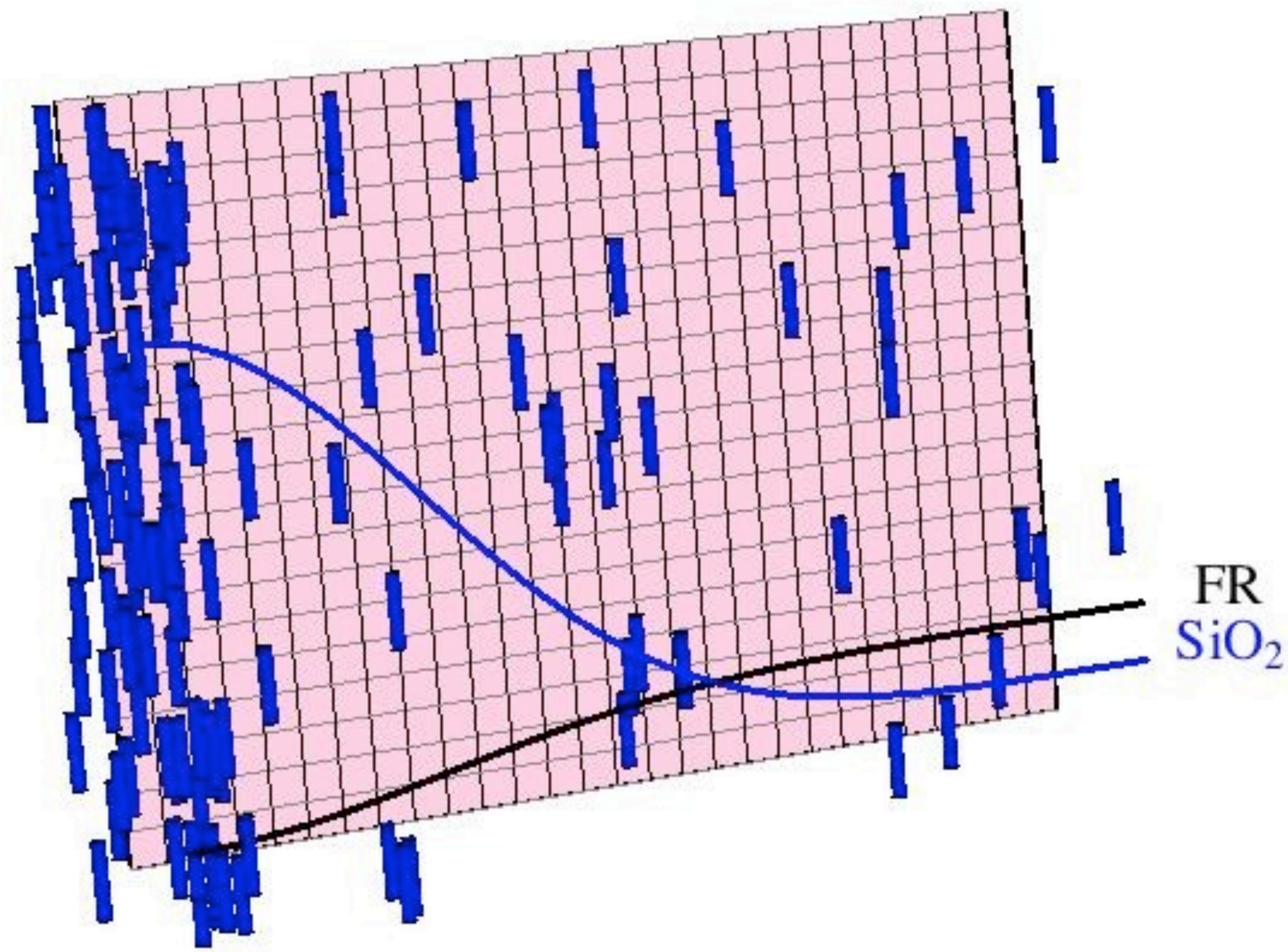
above K-edge

- Least squares fit of X-ray images yields two volumes of data showing BT-93 and Sb_2O_3 concentration distributions. The colorbars show volume percent composition.
- Spatial correlation is obvious. Correlated with mixing order.



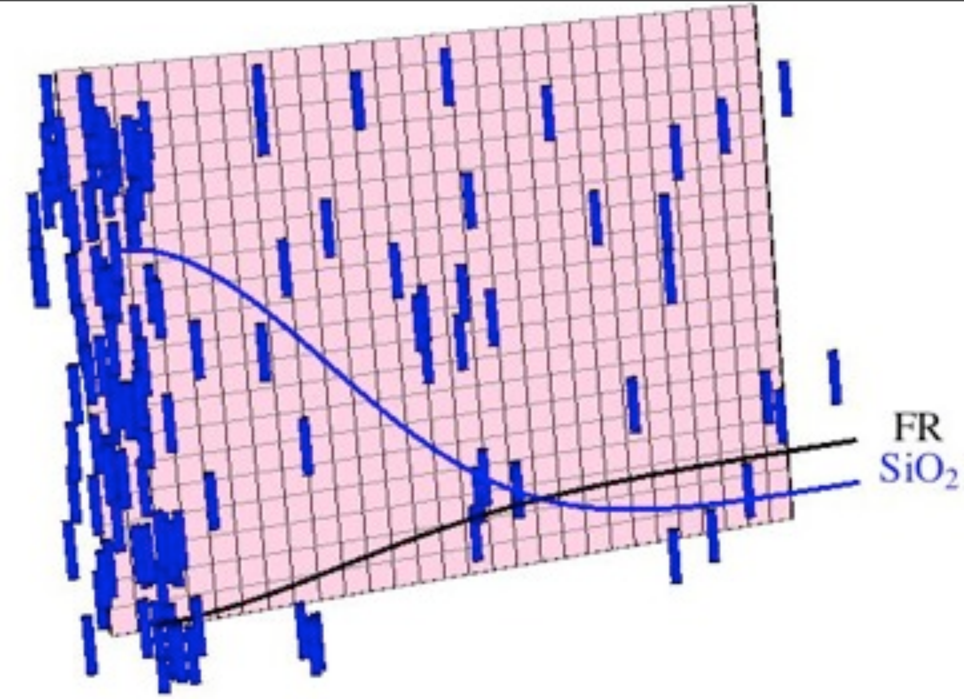
Ham, K.; Jin, H.; Al-Raoush, R.; Xie, X.; Willson, C. S.; Byerly, G. R.; Simeral, L. S.; Rivers, M. L.; Kurtz, R. L.; Butler, L. G. *Chemistry of Materials*, **2004**, *16*, 4032-42.

Sample: Fiberglass reinforced nylon with flame retardant and Sb_2O_3
Questions: What are FR and Sb_2O_3 concentrations around fibers?

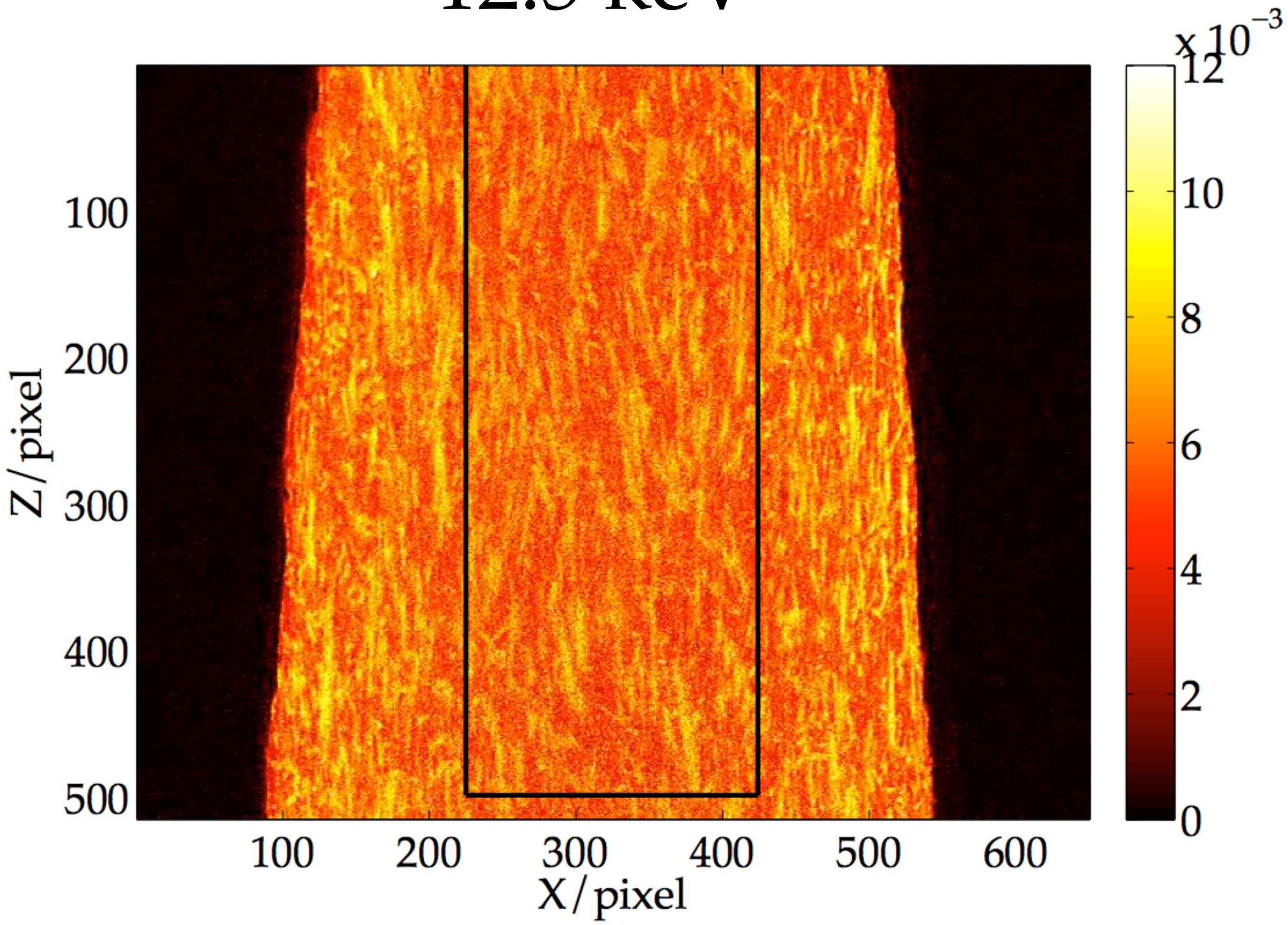


Barnett, H. A.; Ham, K.; Scorsone, J. T.; Butler, L. G., "Synchrotron X-ray Tomography for 3D Chemical Distribution Measurement of a Flame Retardant and Synergist in a Fiberglass-Reinforced Polymer Blend", *Journal of Physical Chemistry B* **2010**, *114*, 2-9.

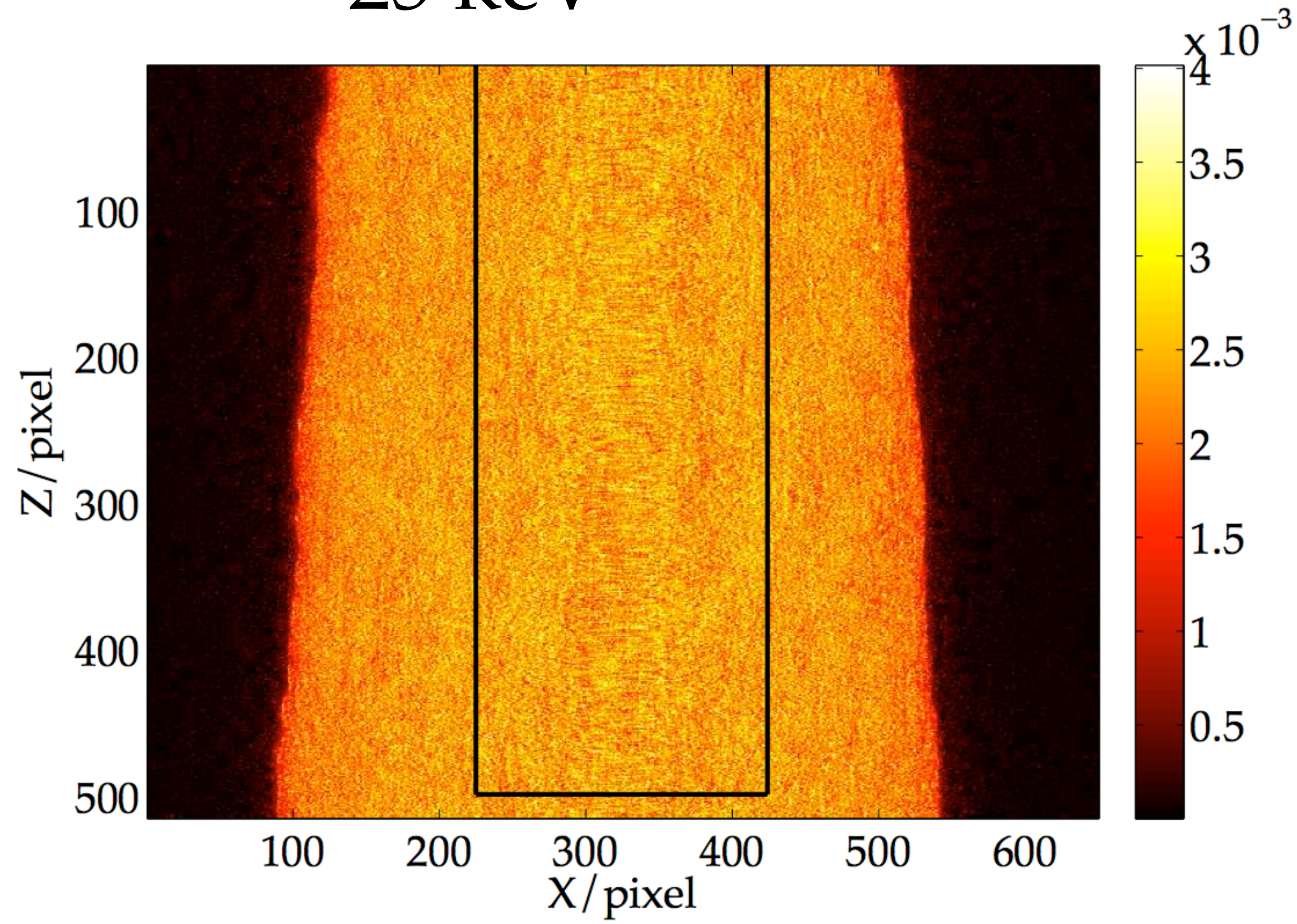
Wow! Structure changes with X-ray energy?
Accidental absorbance matching?



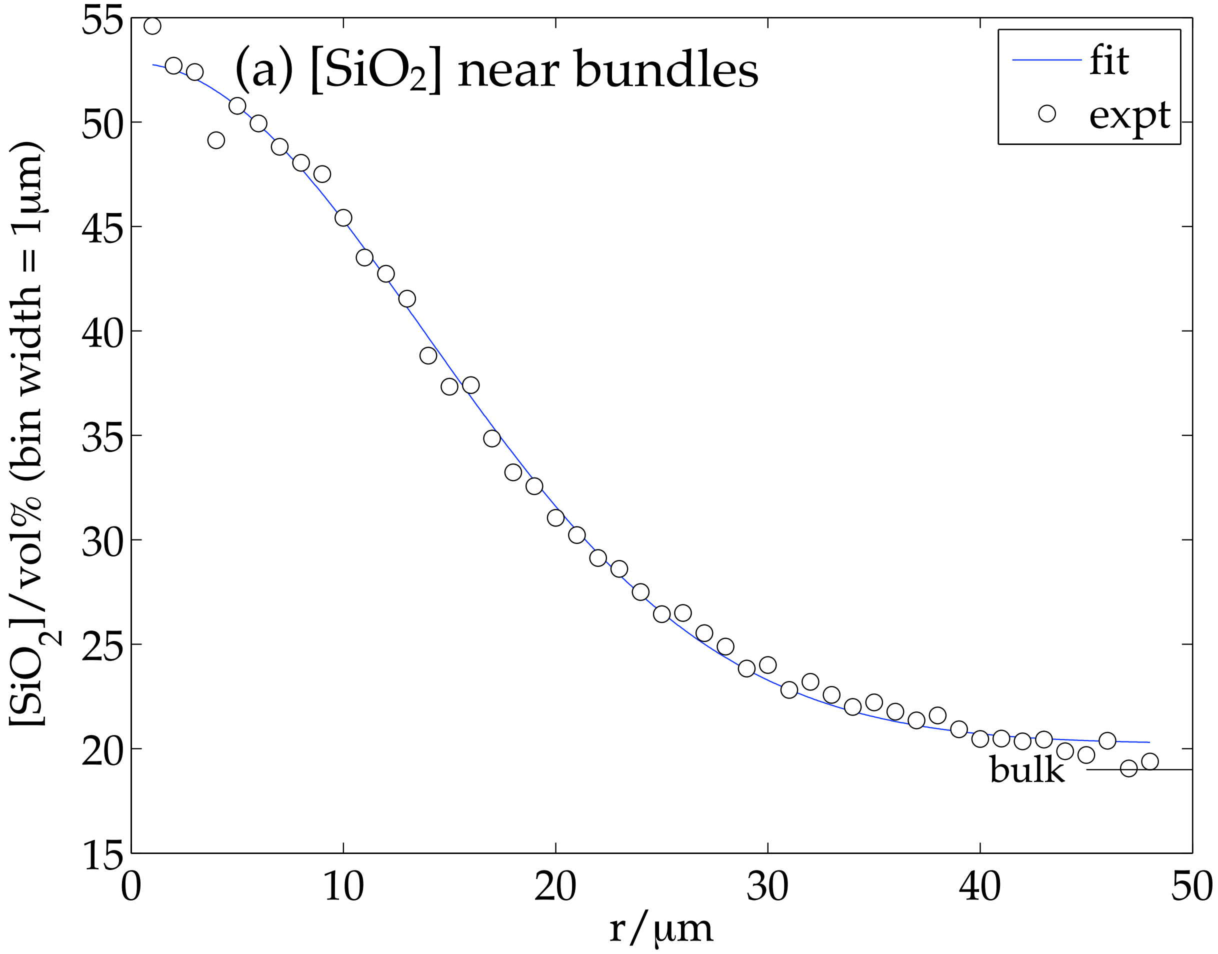
12.5 keV

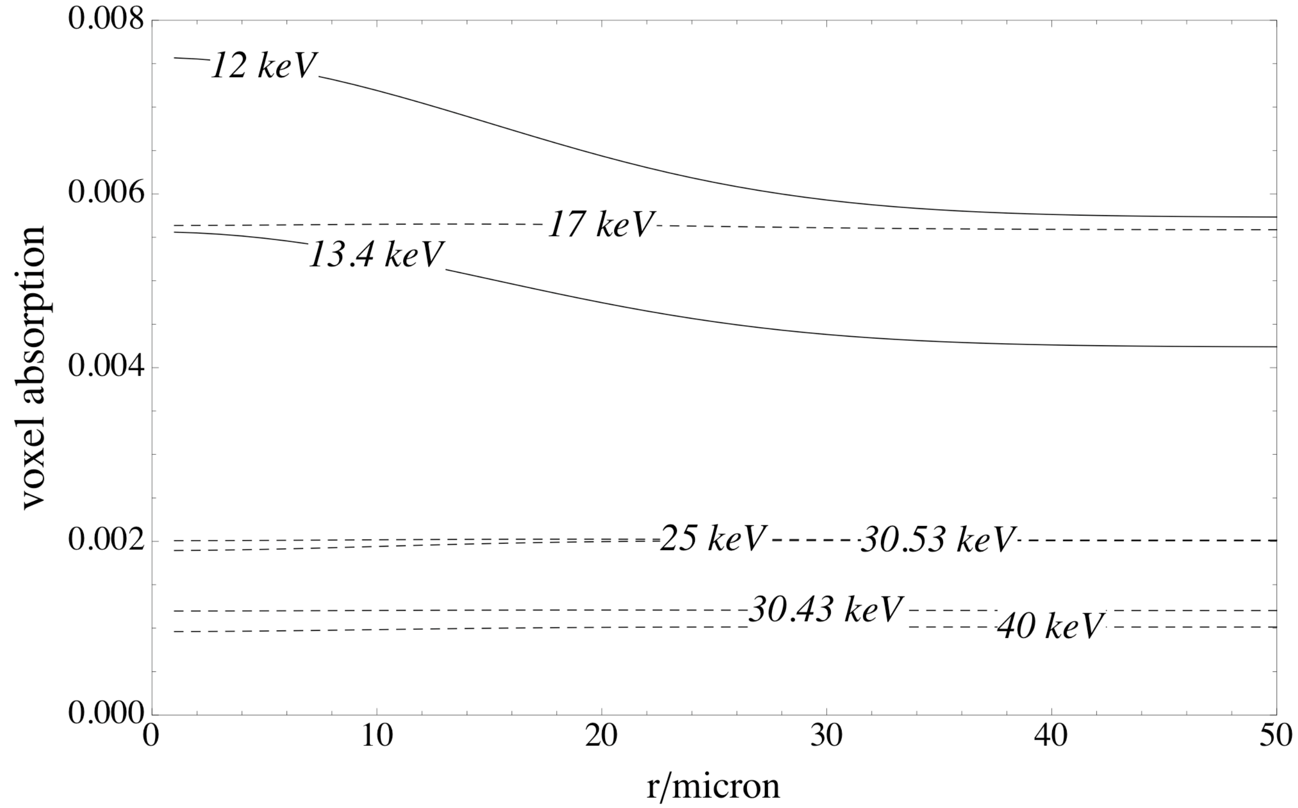


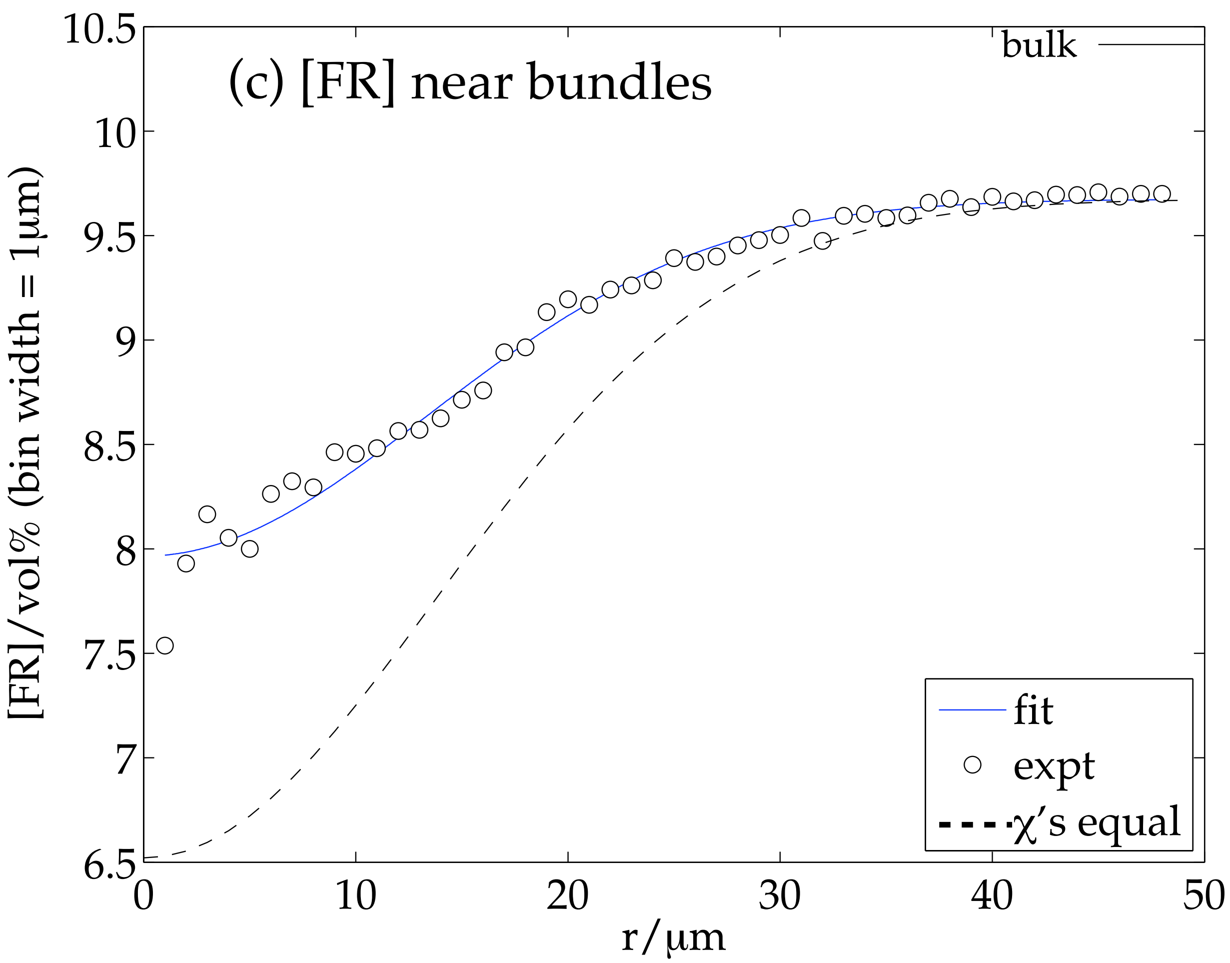
25 keV

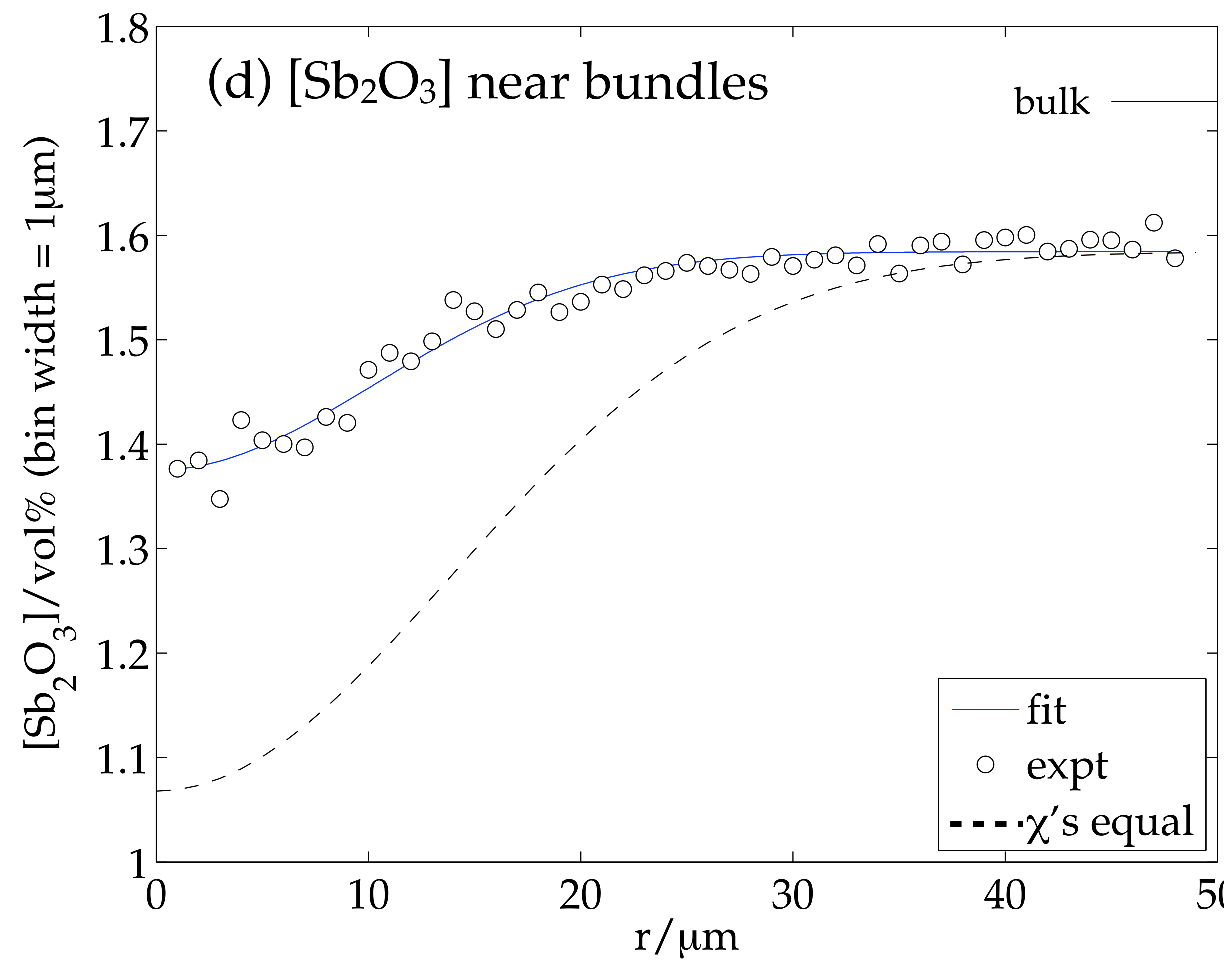


Yes, contrast matching!









Lessons learned

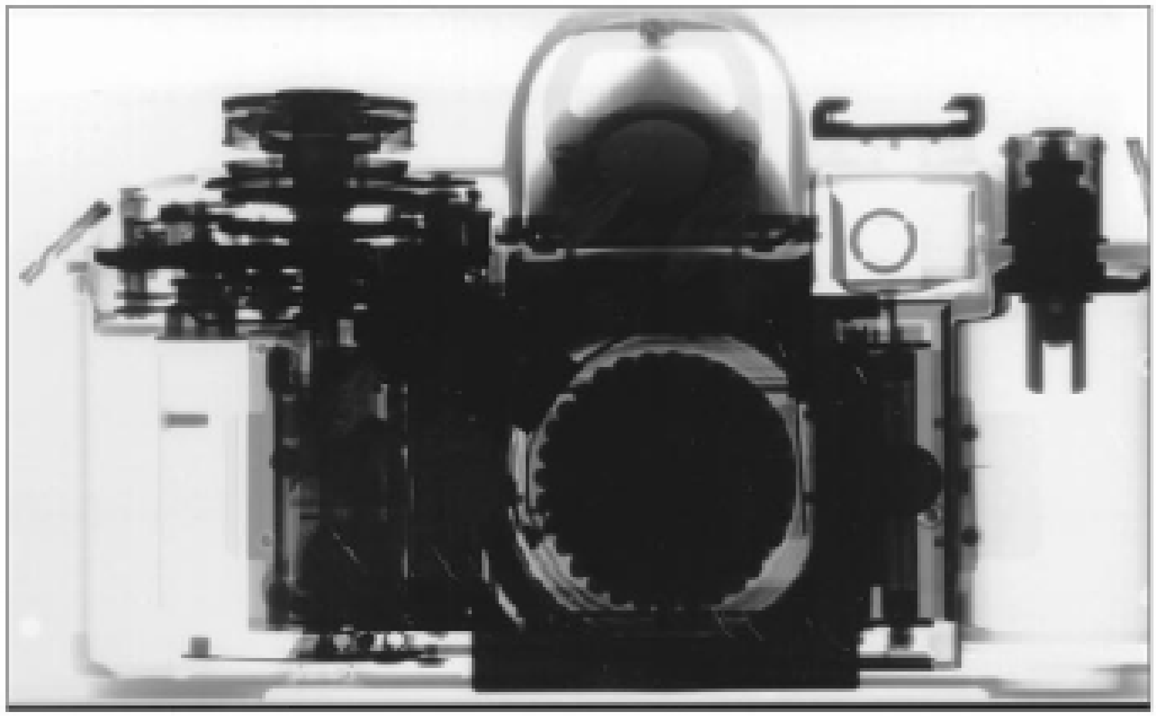
- For samples with a second phase, like a reinforcement fiberglass, have access to a second structure tool like phase contrast imaging.
- Simple phase expts (APS 2-ID-B) don’t work.
- Would like to perform absorption and phase contrast imaging with same sample and mount.
- Expect difficult phase unwrapping problems.

Barnett, H. A.; Ham, K.; Scorsone, J. T.; Butler, L. G., "Synchrotron X-ray Tomography for 3D Chemical Distribution Measurement of a Flame Retardant and Synergist in a Fiberglass-Reinforced Polymer Blend", *Journal of Physical Chemistry B* **2010**, *114*, 2-9.

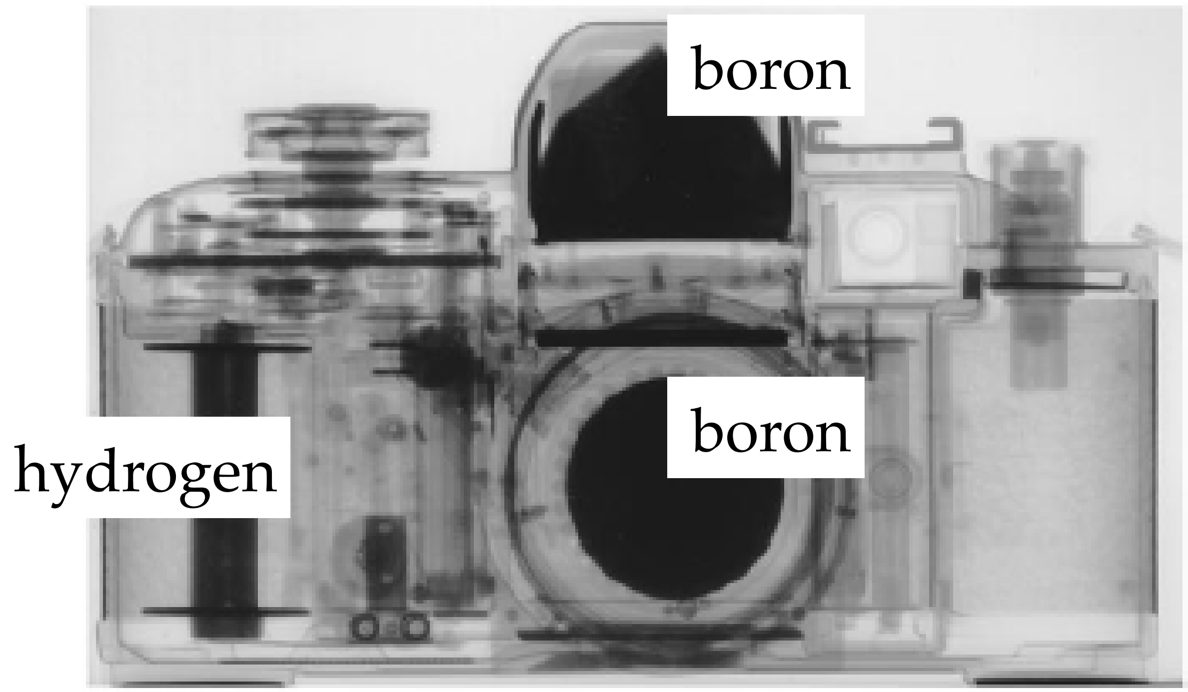

Eberhard Lehmann
Burkard Shillinger
NEUWAVE 1
Munich-Garching, April 20th to 24th, 2008.

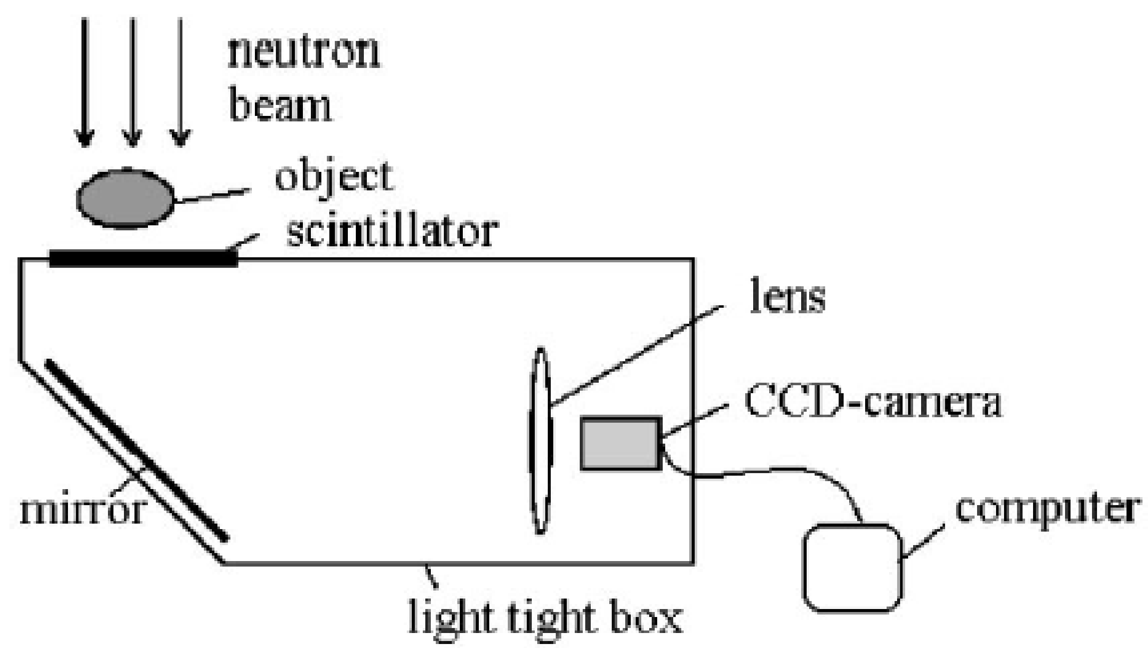
Projections: X-ray vs Neutrons

X-rays



Neutrons

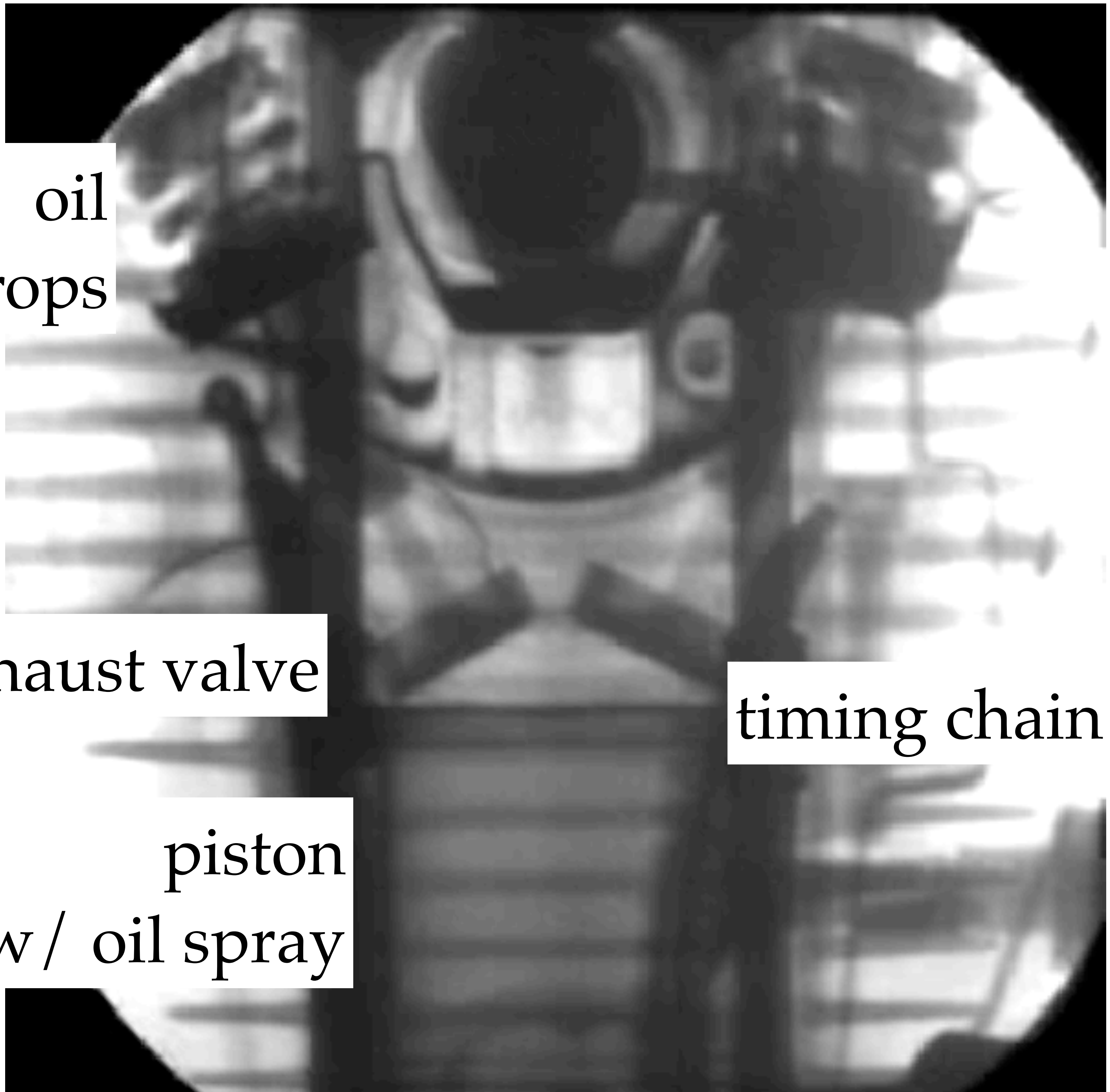




Koerner S.; Lehmann,E.H.; Vontobel, P. Nuclear Instruments & Methods in Physics Research Section a-Accelerators Spectrometers Detectors and Associated Equipment 2000, 454, 158.

Examples of Neutron Radiography in Engineering

- FRM II (Munich): air-cooled gas engine



This work uses back projection.

Plans to use discrete tomography for 3D movies.

Movie recorded at ILL by G. Frei (PSI), B. Schillinger (FRM-II), and A. Hillenbach (ILL), et al.

Spallation Neutron Source (ORNL)

- Neutrons are produced at near-point source by the collision of high-energy protons with a Hg target: 695 ns pulse with 60 Hz rep. rate. Facility costs $1.2B.
- The experimental hall is about 3/4-populated.
  Is there room for tomography?

Linear accelerator

Experimental hall

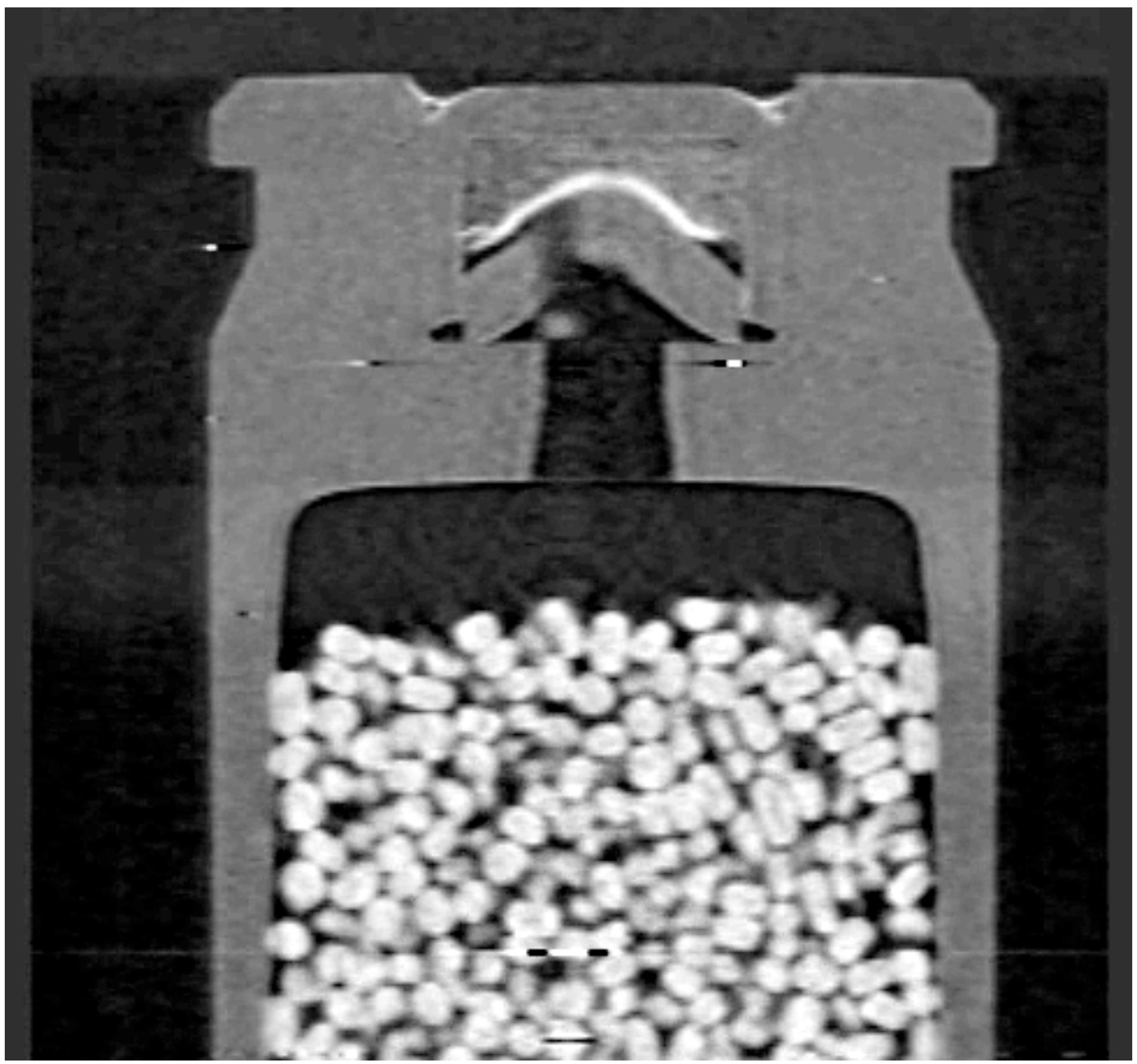
Time-of-flight neutrons expts. radically different from traditional reactor sources.

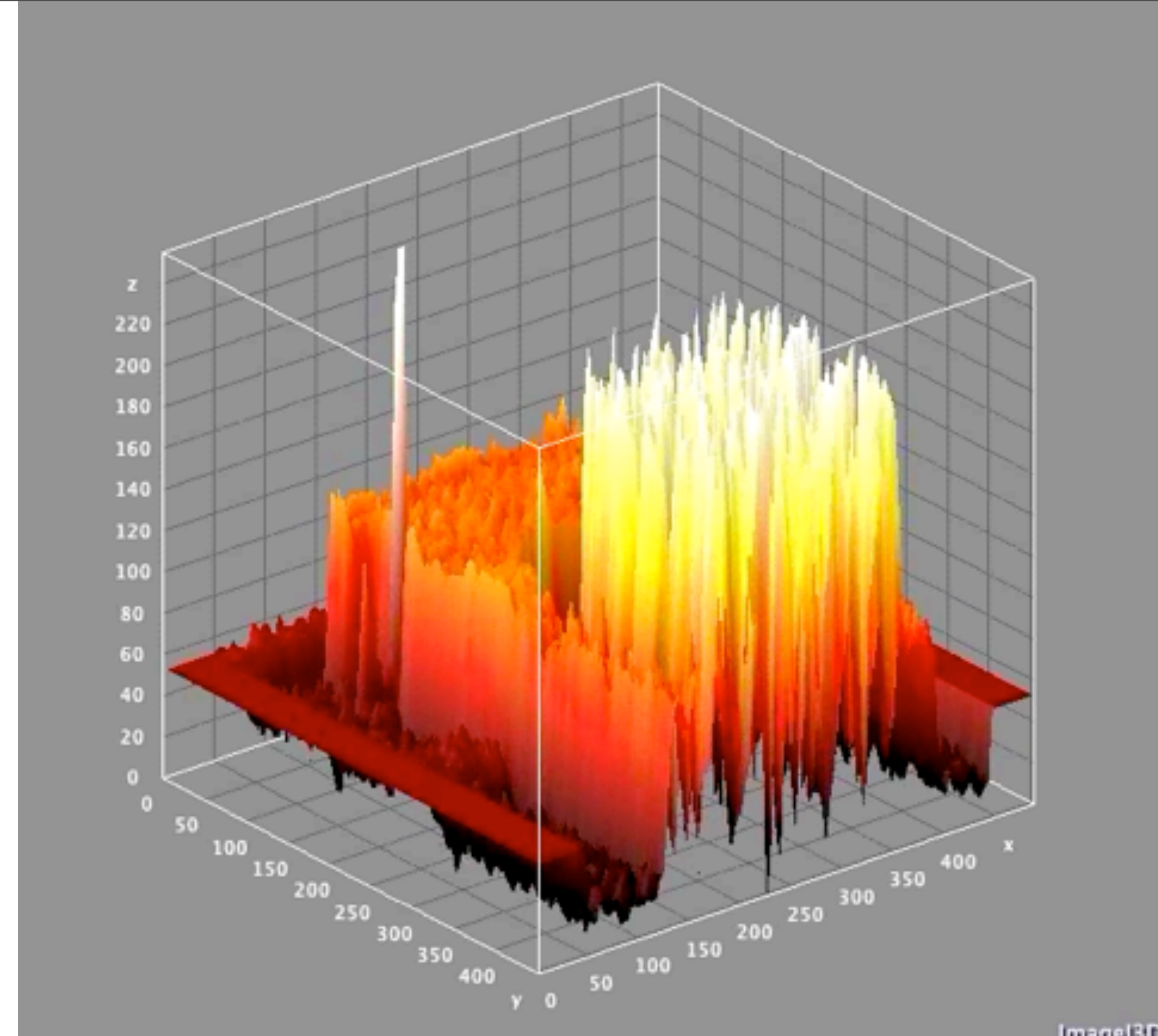
My sabbatical

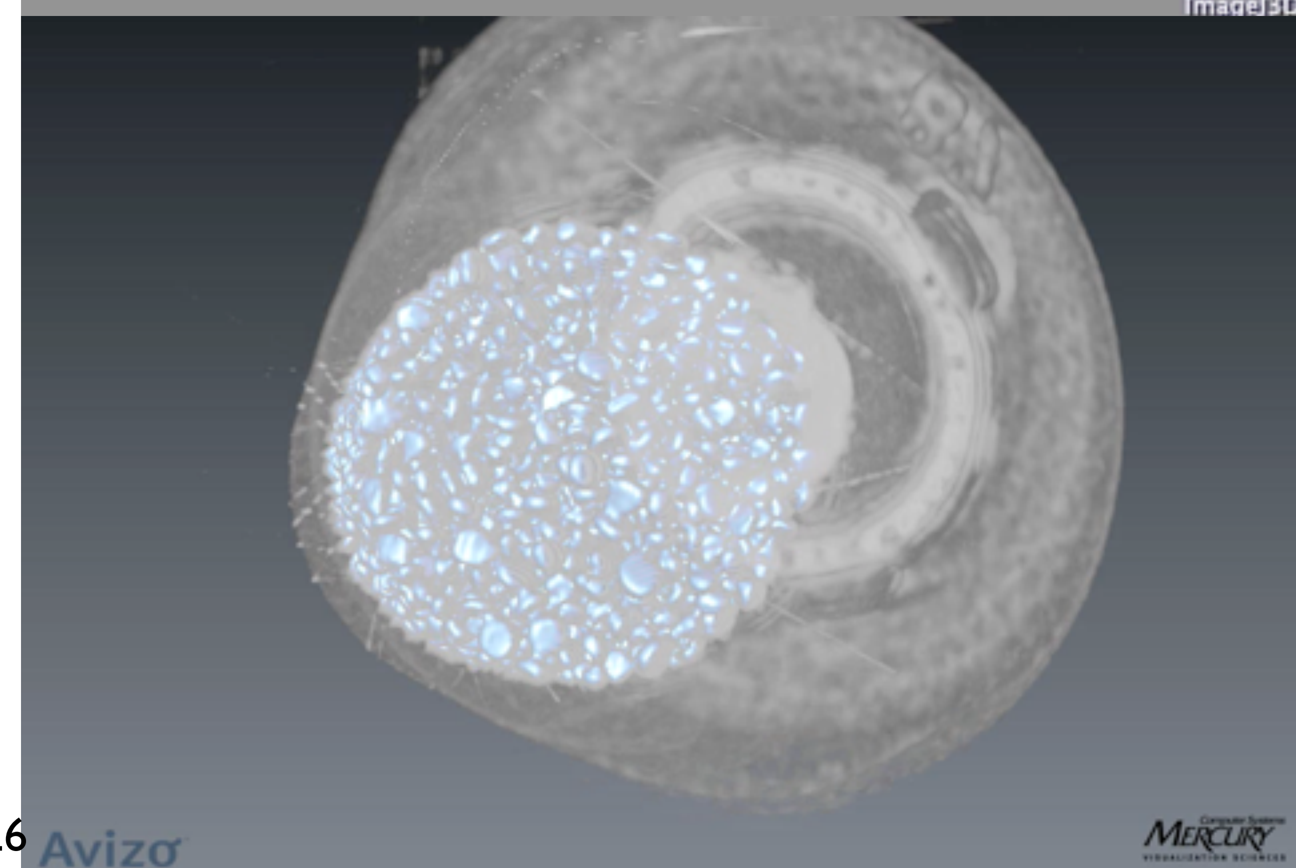
One year scattered across FRM II, PSI, NIST, HFIR, SNS VULCAN.

- Li-ion battery at FRM II
- H_2 storage at NIST
- Imaging artifacts at PSI
- Battery at SNS VULCAN

PSI: Interesting result is that one can image through a metal and see gunpowder, composed of light atoms. This would be difficult with X-rays.







Lithium-ion polymer battery

A Kokam 450 mAhr, two-cell battery was fully charged, then split into separate, but intact cells. One cell was imaged in 2D and the other imaged in 3D. A single cell measured 4.7 mm × 29.5 mm × 43.8 mm with a mass of 13.1 g and a stored energy of 6.0 kJ. Based on the battery capacity of 450 mAh, the battery must contain at least 1.33 g LiC_6 when charged, and then generates about 3.29 g $LiCoO_2$ from Li_xCoO_2 (assume x=0.5) upon discharge.

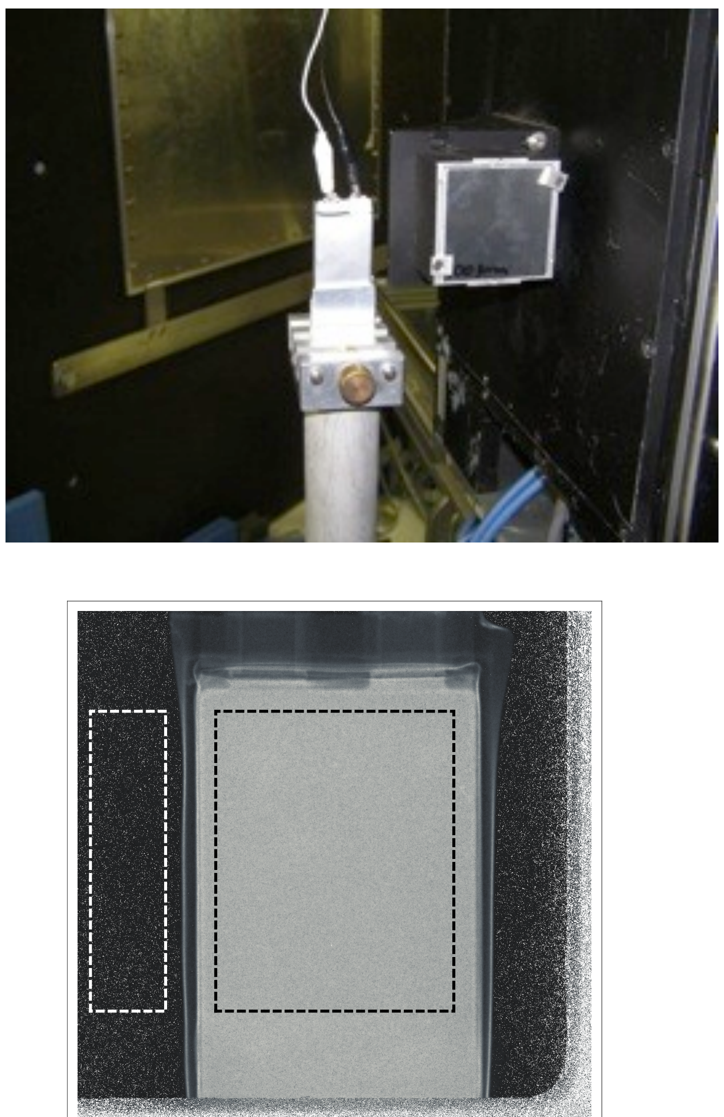
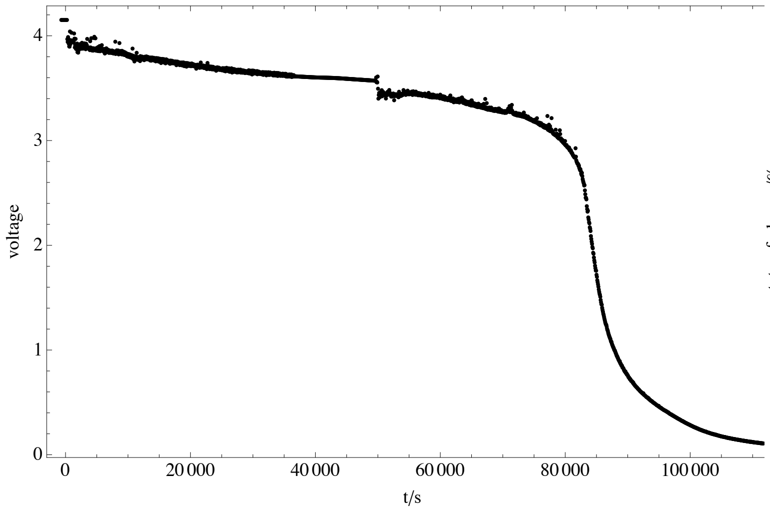
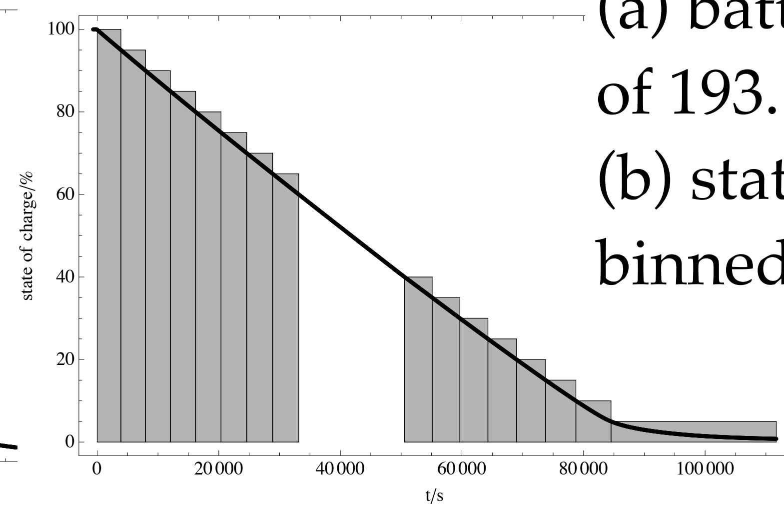


Fig 1. (a) photo of battery mounted on tomography stage and (b) image of battery taken with 3.6 Å neutrons. The white and black dashed rectangles denote image regions selected for averaging the sample and air attenuation values.

work done at FRM II, project ID3202

Lithium-ion polymer battery: real-time 3D imaging with polychromatic neutrons



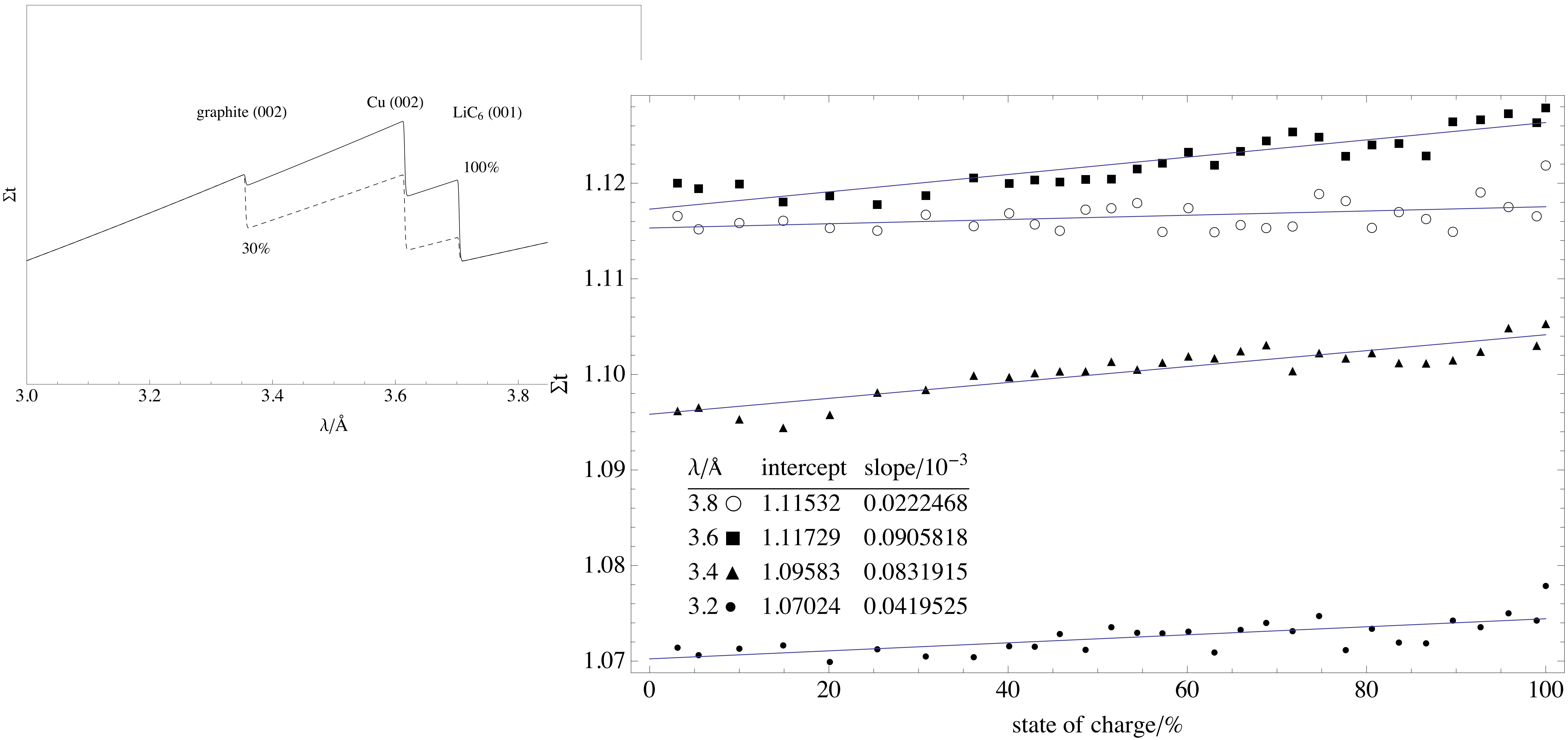


(a) battery voltage vs. time for a resistive load of 193.1 Ω and
(b) state-of-charge vs time for the projections binned to generate 3D images.

SOC = 100% 95% 90% 85% 80% 75%

SOC = 30% 25% 20% 15% 10% 5%

Lithium-ion polymer battery: real-time 2D imaging with monochromatic neutrons



This result funded 14 more days of neutron beamtime at PSI, FRM II, and SNS VULCAN.

SNS VENUS: status

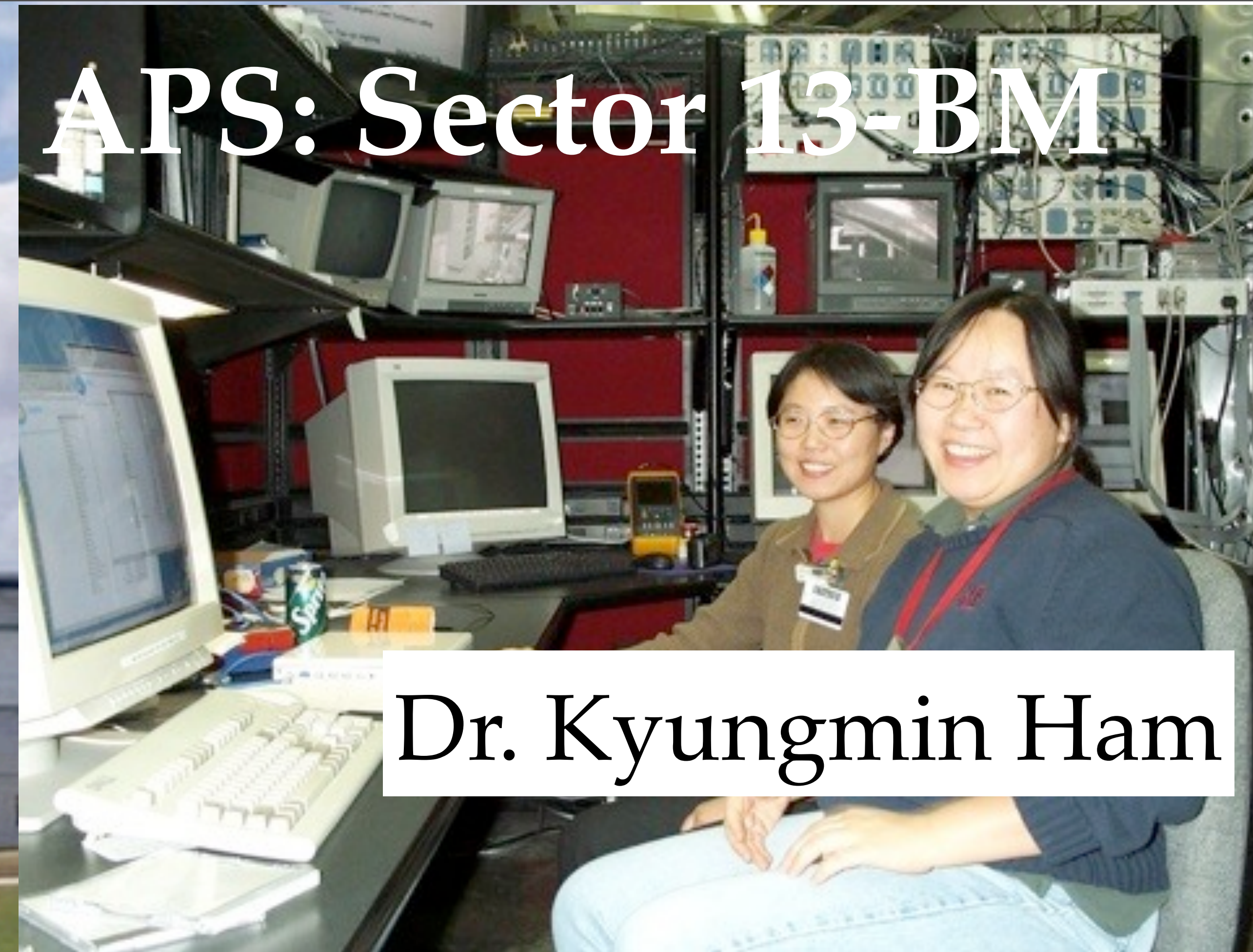
As yet (June 2010), there is not a tomography beamline at the SNS. Many steps needed to get a new beamline. In brief, these steps are:

1. Workshops
2. Preproposal: approved, Nov, 2008
3. Full proposal to neutron science advisory committee (to come). **Clock starts with approval and port allocation.**
4. Concept & engineering design funds, ~$2M. Proposal under review (May 2010).
5. Construction funds, ~$15M. Proposal not yet started.

Dynamic Imaging with Phase and Absorption Contrast (DIP)

by Les Butler
Department of Chemistry
Louisiana State University

- large user community from GeoX 2010 workshop:
  - fantastic research, great ideas, really nice people!!!
- personal research:
  - flame retardants in polymer blends
  - neutron tomography: co-champion of SNS VENUS project
  - LSU CAMD synchrotron tomography beamline: testbed?
  - IMA math workshop
- wish list for NSLS II imaging beamline(s)



Can LSU CAMD tomography serve as a testbed for NSLS II projects?

- 2009: NSF MRI grant funded for $1.26M for improved X-ray flux (wiggler).
- We can leverage the NSF grant into more state funding for equipment at tomography beamline. What should we get? Phase gratings, etc?

LSU synchrotron: CAMD

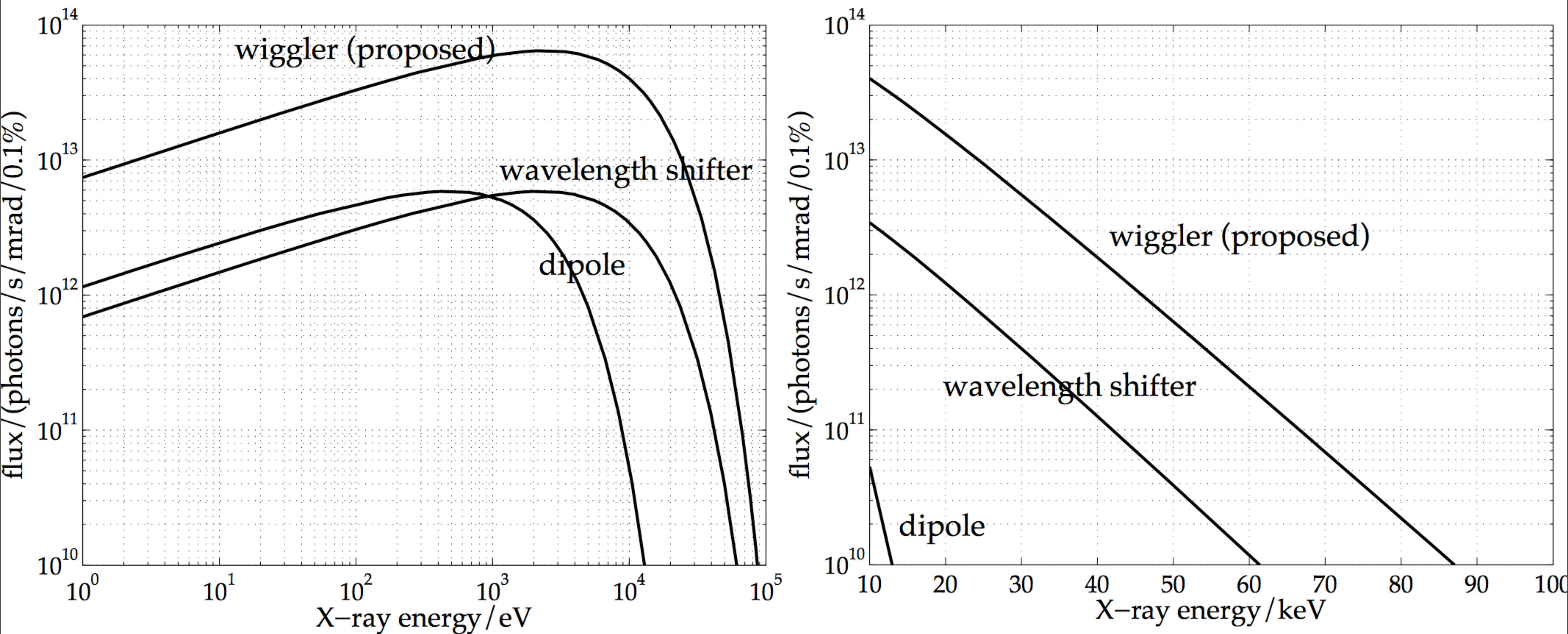


Figure 5. Calculated photon flux from proposed 11-pole, 7.5 T wiggler *vs*. current wavelength shifter and bending magnets at CAMD. Important for protein crystallography is the 12-fold increase at 12.66 keV (Se K edge). Tomography will see 12- and 15-fold increases at 13.47 and 33.17 keV (Br and I K-edges).

Mathematics: reconstruction and 3D image analysis

Jan 9-12, 2006 in Minnesota
http://www.ima.umn.edu

IMA - Institute for Mathematics and its Applications

Imaging, September 2005-June 2006

IMA Workshop:

3-D Image Acquisition and Analysis Algorithms

January 9-12, 2006

Organizers:

Les Butler
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Dining Guide			Maps

Schedule and list of participants are not yet available.

New mathematics and algorithms are needed for 3-D image acquisition and analysis. The 3-D images come from many disciplines: biomedicine, geology, chemistry, and microfabrication. The mathematics is wide-ranging and includes at least tomography and inverse problems, wavelets, PDE, and conformal mapping. The depth of the problem and the extent of the mathematics argues for multiple, long-duration collaborations that are fostered by a workshop series.

Wishlist

1) X-ray energy to 100 keV

2) quick switching from about 5 keV to 100 keV (like automatic switching of two monochromators)

3) maybe an insertion device, but maintain the phase coherence

4) and option for just under 1 micron resolution. Better yet, automatic switching from 5 micron to 0.2 micron

5) remote control

6) phase acquisition in some mode such that complex phase properties can be unwrapped.

7) acquisition such that same sample can be imaged with absorption and phase (goes with option 2 above)

8) rapid acquisition mode, like 1000 frames/second with good white frame control

9) sample stage can handle clear field-of-view load cell to a pressure of 10,000 psi or something on the order of 170,000 Newtons over a (5 cm)^2 area.

10) rotation axis align along vertical or horizontal (100x better phase coherence in vertical direction)

11) local computer cluster with several processing languages.

12) More math workshops